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# Report on a Trial Challenge Inspection in a Chemical Industry Plant

## Introduction

In a continuing effort to foster the conclusion of the chemical weapons convention and to gather experience and technical knowledge for the future implementation of the convention, a trial challenge inspection has been conducted in a chemical industry plant in the GDR. The plant selected was the WOFATOX-factory of Chemiekombinat Bitterfeld (Chemical Combine Bitterfeld), a facility producing the organophosphorous pesticide parathion-methyl.

This is a general description of the activities undertaken and of preliminary conclusions drawn. The report is in four parts:

1. The aim of the trial inspection and its basic approach
2. Description of the inspection methodology
3. Experimental validation of the inspection methodology in laboratory experiments and in the actual trial inspection
4. Summary and preliminary conclusions

More specific information on inspection methodology and trace analysis is included in two related working papers.

## 1. The aim of the trial inspection and its basic approach

The aim of the trial has been to develop and evaluate an inspection methodology for challenge inspections in industrial plants, and to improve the understanding of the technical implementation of Ad-Hoc-type inspections.

Inspection procedures and time frames were not the primary targets of the trial. Nevertheless, some preliminary conclusions could also be drawn on these issues. At the core of the experiment, however, were methodical problems of the conduct of challenge inspections in industry plants. This may be of some relevance for the elaboration of an inspection manual.



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The basis for the trial has been the following mandate:

"Verify whether or not at the WOFATOX-plant, part of the larger industrial complex of Chemiekombinat Bitterfeld and located in Wolfen, GDR, any organophosphorous chemical listed under schedule 1 has been produced".

The inspection aim was thus not limited to verification of compliance at the time of inspection. An unspecified retrospective compliance validation was also requested. Verification results would have the nature of a probability statement with decreasing reliability for increasing retrospective time coverage.

The approach designed for the trial challenge inspection was a "layered inspection methodology" (i.e., a step-by-step approach) with increasing intrusion triggered by the results of the less-intrusive inspection layers (phases) together with an assessment of these results in the light of the circumstances and particularities encountered. That was considered to allow an inspection team to develop its actual inspection strategy depending on the specific situation at the site. Four such inspection layers (phases) were designed. Consideration was also given to the possibility that the plant management, in case that treaty provisions had been violated, had implemented a counter-inspection strategy in order to cover-up clandestine activities. It was assumed that the inspectorate would have no specific beforehand knowledge on the plant to be inspected.

In the actual inspection experiment, only some crucial increments of the methodical concept were in fact put to test while others were validated theoretically.

## 2. Description of the inspection methodology

The detailed methodical approach is described in another working paper. In the following, the aims of the four phases are briefly described.

### Phase 1

Detection and identification of chemicals listed under schedule 1 at the inspected plant pursuant to the inspection mandate (i.e., organophosphorous schedule-1-chemicals in this trial inspection).

This phase was considered mandatory for any challenge inspection in an industrial plant. It was also considered to correspond to the aim of an Ad-Hoc-type inspection.

### Phase 2

Assessment of the plant with the aim to conclude whether or not it may pose an immediate and high risk to the objectives of the convention. Risk in this context was understood to comprise both the potential CW capability of the plant and its roughly estimated capacity in case it was assessed

capable. CW capability was defined for the purpose of this trial inspection as being made up of the following increments: "chemical capability" (i.e., the availability of requisite chemicals), "technological capability" (i.e., the availability of requisite equipment), safety features, and the overall security regime at the inspected site. A part of this phase would also be to confirm that no signs are present which might be interpreted as residues of cover-up activities at the inspected site.

**Phase 3**

Resolution of any anomalies which have been encountered in phases 1 and 2 with the aim to allow to conclusively demonstrate compliance with treaty provisions pursuant to the inspection mandate.

In case that not all anomalies can be resolved and that the inspection team assesses an immediate and high risk that treaty provisions may in fact have been violated, the inspection would continue with phase 4.

**Phase 4**

Highly intrusive inspection activities in order to conclusively demonstrate compliance, or to prove a violation of treaty provisions.

The basic approach in any of these inspection phases was to request specific facility statements and supportive documentation. The content, validity, and truth of these statements was then to be verified to the extent possible.

## 3. Experimental validation of the inspection methodology in laboratory experiments and in the actual trial inspection

### 3.1. Laboratory tests and other pre-inspection activities

#### 3.1.1. Development of analytical methods

The analytical methods used for the trial challenge inspection were ion mobility spectrometry and gas chromatography. Transportable instruments were used in either case.

While IMS was applied for trace detection and identification of organophosphorous schedule-1-chemicals, a combination of portable GC and IMS was applied in order to validate the facility statement about the plant's actual production activities.

Schedule-1-chemicals were simulated by a nerve agent simulant having a proton affinity almost the same as that of nerve agents: diisopropyl-methylphosphonate (DIMP).

Sampling techniques and analytical methods were developed and tested in the laboratory in order to prepare for the on-site analysis of different types of samples. These, together

with corresponding laboratory results are presented in another working paper. The following conclusions were drawn for the analytical approach of the trial inspection:

1. Trace identification of the simulant used in the study was possible against a background concentration at least three orders of magnitude larger than DIMP of a number of organophosphorous pesticides.

2. Traces of the simulant remain detectable, and can in fact be identified, in material typically used as joint packing, for at least 580 hours. A method was designed to analyze DIMP traces from joints without braking them, by sucking-off ("sniffing") air from the head-space around the joint packing.

3. In collecting wipe samples from metal surfaces, approximately 1 microgram DIMP can be detected and identified against a pesticide background concentration as above.

### 3.1.2. Risk assessment of the pesticide plant

A methodology for risk assessment was developed in order to provide the inspection team with an objective guidance in assessing whether or not the facility may pose an immediate and high risk to the objectives of the convention, by verifying the absence or presence of unusual features at the plant.

The pre-inspection activities encompassed the feasibility testing of the application of a computer program for evaluating the "chemical capability" of the inspected plant. This computer program had originally been designed for generating synthesis strategies based on synthone substitution[1].

Conceptual preparations were conducted for the other elements of the risk assessment. These elements were:

- Qualitative assessment of the territorial features at the inspected site in order to evaluate the potential down-wind hazard for nearby inhabited areas.

- Qualitative assessment of the safety precautions taken at the site in order to identify or demonstrate the absence of unusual (as compared to best international practice in industrial plants) preparations for rapid decontamination, for protection of personnel, and/or for early warning.

- Assessment of the installed technology and equipments in order to identify or demonstrate the absence of special equipments necessary for the production of schedule-1-

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[1] The program and its potential applications for verification purposes will be described in more detail in a later working paper.

chemicals which otherwise have rare or no application in the type of plant inspected.

- Assessment of the overall security regime at the plant, in order to identify or demonstrate the absence of security features irregular for industrial plants.

### 3.2. The actual conduct of the trial challenge inspection

The inspection was conducted at the end of march 1990. It had been preceded by a number of in-plant tests to validate analytical techniques. What follows is a concise account of inspection activities carried out, and results thus achieved.

The inspection team consisted of:

- 1 team leader
- 4 analytical chemists
- 1 chemical engineer

For real inspections, it was assumed that additional team members might become necessary (e.g., one more chemical engineer with experience in organophosphorous chemistry, two inspectors experienced in accounting, and several more team members to secure the site).

The plant was represented by the facility operator and a representative of the management of the chemical combine. In the exercise, representatives of the Ministry of Foreign Affairs and the Ministry of Disarmament and Defence participated as observers.

Several procedures and inspection elements of a challenge inspection were not actually conducted in the trial. The initial briefing was not in fact carried out in the actual trial itself, but was an element of the pre-inspection preparations to develop technical methods and concepts. The trial took one day, not counting pre-inspection activities.

#### 3.2.1. Analysis of environmental and other samples immediately after arrival at the plant

The team conducted a quick visual tour of the site and selected the following points for prompt analysis:

- Air samples: taken outside the plant building, inside the building, and close to reaction vessels, joints, temporary storage tanks, etc.;
- Wipe samples: taken from reactor surfaces close to valves, joints, and reactor in- and outlets

No IMS signals of the simulant used (or in fact of any other chemical listed under schedule 1) were detected in the samples. Proper functioning of the instrument was confirmed by parallel checks with insertion of a diffusion tube containing DIMP into the sampling pipe. The method applied for sampling had been validated beforehand in laboratory

experiments, as had been the possibility to identify residues at trace level after prolonged periods of time.

### 3.2.2. Request of a facility statement on the non-production of schedule-1-chemicals and subsequent verification

The manager was requested to declare whether or not the plant has produced, or had done so in the past, any of the chemicals listed under schedule 1. After compliance with that request, he was asked to furnish a technological scheme showing the lay-out and pipe-network of the plant in order for the inspection team to identify relevant sampling points for chemical analysis. That scheme also included a list of installed equipment.

The details of sampling points so selected, the justification of that selection, and the corresponding analytical results are described in another working paper. The results were in conformity with those achieved in the initial checks described above. It is worth mentioning here that for the analytical technique used to verify the absence of organophosphorous schedule-1-chemicals (IMS), sample duplication was usually not considered feasible because the measurement was typically performed by head-space sampling directly into the instrument inlet system. Sample duplication could, however, be done in case of positive signals when additional samples would have to be taken for confirmation analysis by an independent trace-analytical method (e.g., mass spectrometry) preferably to be carried out at a stationary laboratory. Another feature to be mentioned here is that samples were in most cases collected by the inspectors themselves given the nature of the sampling techniques applied (wipe samples, dust samples, air samples, sniff-test). The exception was a sample taken from the interior of a reaction vessel. That sample was taken by the facility operator on request of the inspectors and under their supervision.

The facility management was also requested to declare the actual production at the plant and the chemicals present at the site as starting materials, intermediates, and final products. These data were confirmed by IMS and GC analysis at points selected by the inspection team.

### 3.2.3. Risk evaluation

The facility management was requested to provide answers to the following questions, and submit the following documents/materials:

- state the internal volumes of the production units (reactors, storage tanks for final product and intermediate chemicals, etc.);
- declare whether or not the plant is separated (physically, separate supply lines for chemicals, separate waste/effluent treatment, etc.) from the remaining complex;

- declare whether or not the plant has taken special precautions against chemical accidents, and describe these in a general way if applicable;
- declare whether or not the plant uses or produces chemicals listed under schedules 2 or 3 of the convention; If so, which chemicals are used, for which purposes, and how much per annum;
- declare whether or not any of the following equipments is installed in the plant: (the list used for this purpose was drawn from annex 2 of the report on the National Trial Inspection of Australia, CD/910 of April 4th, 1989, page 21), and give a justification if applicable.

In the further course of the inspection the following activities were undertaken:

1. Computer evaluation of the "chemical capability" of the inspected plant

A computer program was used to assess whether or not a production of schedule-1-compounds is chemically possible with the material present at the plant. Given the nature of the plant and the chemicals used there, the expected positive result was computed.

Yet, there was no synthetic path computed with the chemicals (starting chemicals, intermediates, final products) available at the plant which would have allowed to produce a schedule-1-chemical in a one- or two-step synthesis and without access to additional chemicals not present at the site during the inspection. Some of these chemicals might, of course, be accessible from other factories at the complex. This was not however verified during the inspection.

2. Visual inspection of the plant

A close visual inspection of the plant was conducted in order to qualitatively verify data provided by the management of the factory and to gather relevant information for completion of the risk assessment as briefly described above. The following observations were regarded relevant in this respect:

- The plant has no automatic alarms or sensor arrays for highly toxic chemicals installed.
- The plant is using equipment which would not have allowed to provide the leakage-tightness necessary to produce supertoxic-lethal chemicals if plant personnel was not operating under full protection (also validated by analytical results in the plant area).
- The plant's waste-water treatment system would be unsuited for cleaning-up waste streams from nerve-agent production.
- The plant's effluent-gas cleaning system was also considered unsuited for the purposes of nerve agent production, a usual reactor ventilation system is operated.
- The equipment used at the plant would not allow to process highly corrosive materials (such as HF).

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- No preparations were detected for a rapid clean-up or decontamination of the factory area or the interior of the plant.
- On-site preparations for chemical accidents (emergency gas masks etc.) were those normally to be expected at an industrial chemical plant of the type inspected.
- The plant was not separated from other factories of the complex (i.e., no plant-specific security regime, no separate supply lines or waste/effluent transportation and treatment).

3. Given all these data, the inspection team concluded that the plant's chemical supply, technological and safety lay-out, and security regime were not to be considered well-suited for a production of organophosphorous schedule-1-chemicals without major modifications. It does not pose an immediate risk to the objectives of the convention.

**3.2.4. Inspection activities in order to rule out attempts to cover-up previous activities by plant clean-outs or modifications**

As a basis for a close visual inspection in order to confirm the absence or detect the presence of attempts to cover-up prohibited activities at the factory, the inspection team could make use of the most recent revision of the plant schemes available which dated from 1984. Several minor modifications had been implemented in the meantime without being recorded in these technological schemes.

The purpose of that visual inspection was to find traces of recent modifications and/or clean-up activities at the site (e.g., residues of extensive cleaning operations, of decontamination agents suitable for nerve agent detoxification, visible signs for recent replacements of certain equipments, signs for replaced joint packings, etc.). There were no signs of unusual and extensive recent cleaning or decontaminations operations in the plant area and on surfaces of reactors and other equipments. No other signs/traces which could be interpreted as residues of cover-up attempts were detected. Given the time frame presently considered for challenge inspections and the extensive effort which would have been necessary to clean-out, decontaminate and re-start the inspected factory, the members of the inspection team felt absolutely confident in their conclusion that such attempts could be ruled out.

**3.2.5. Inspection results**

1. At the time of inspection, no traces of schedule-1-production could be detected at the site. The team's ability to do so was demonstrated by validation tests using the model compound DIMP. A methodology was applied to exploit memory effects in a plant in order to identify, at trace level, former production activities.

2. An assessment was conducted of the plant's potential risk to the objectives of the convention using a combination of different methods. Without modifications, the plant was found not to pose an immediate/high risk to the objectives of the convention.

3. The plant was scrutinized for traces of recent clean-outs or replacements in the technological lay-out which might point to cover-up attempts. No such traces were detected. No further inspection activities were considered necessary in order to conclude the inspection.

## 4. Summary and preliminary conclusions

1. A phased (layered, step-by-step) inspection approach for challenge inspections in industrial plants has been designed and partly tested which, in the assessment of the participants of the verification experiment, would allow for a sound conduct of such an inspection based on the principle of using the least intrusive verification methods possible. The participants in the exercise strongly felt a necessity for an as flexible approach as possible for the conduct of challenge inspections in industrial plants.

2. It was possible in laboratory experiments and in the actual inspection to demonstrate the feasibility of exploiting memory effects in a chemical plant in order to identify residues of former production at trace level. Contamination can be expected at certain surfaces, in so-called dead-spaces in the plant, in materials with a certain tendency to adsorb or absorb organic chemicals such as those typically used as joint packings, or in structures having a certain porosity such as concrete, dust, etc.. Analysis can be carried out in a non-intrusive way that would usually not interfere with normal plant operations.

3. A concept was tentatively developed to qualitatively evaluate the risk that a facility might pose to the objectives of the convention. The concept was successfully applied in the trial in order for the inspection team to decide on the level of intrusion actually necessary for implementing the inspection mandate.

4. Based on the experience gathered in the inspection, the participants of the trial inspection concluded that sufficient time should be available for an inspection team to familiarize itself with the basic lay-out and essential details of a plant in order to identify valid sampling points and to properly plan other inspection activities. This has to be taken into due account in the design of inspection procedures and time frames of Ad-Hoc as well as challenge inspections in industrial plants.

5. At several stages during the inspection, the inspectors had to verify information which the facility management considered confidential. This was especially true of some quantitative data used in the risk evaluation of the plant design and of some details in the facility schemes. Yet, so

far designed procedures to protect confidential information were generally felt to be sufficient to protect unauthorized access to such data. Some of the information so categorized as confidential would not have to be incorporated into the inspection report, and would have to remain on the facility premises.

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# Inspection Methodology for Challenge Inspections in Industrial Chemical Plants

## Introduction

An important principle for the conduct of challenge inspections is for the inspectors to apply the least intrusive verification methods possible while retaining the capability to collect all information that is required to demonstrate compliance or non-compliance. Approaches of implementing this principle are, e.g., the concepts of "managed access" and of "alternative measures". In this paper, another such approach is presented for consideration: a layered (or phased, step-by-step) inspection approach. This approach was developed and partly tested in a trial challenge inspection at an industrial plant carried out in the German Democratic Republic. A report on this trial has been submitted in another working paper. This working paper is a more detailed discussion of the concept of a "layered inspection methodology".

The "layered inspection methodology" consists of four phases with increasing intrusion triggered by the results of the less-intrusive inspection phases together with an assessment of these results in the light of the circumstances and particularities encountered. That was considered to allow an inspection team to develop its actual inspection strategy depending on the specific situation at the site. Consideration was also given to the possibility that the plant management, in case of a violation of treaty provisions, had implemented a counter-inspection strategy in order to cover-up clandestine activities.

In the following phase-by-phase description of the concept, several elements and procedures of a challenge inspection (such as initial briefing, location detection, site securing, etc.) are ignored. For a description of a full-scale inspection, these missing elements and procedures would have to be added.

1. Phase one

Based on the inspection mandate, the aim of this phase is to confirm the absence or presence of schedule-1-chemicals. Prompt analysis may be performed by inspectors of environmental and other samples at the outset of the inspection,i.e. at a stage where the inspection team has only a very limited understanding of the specifics of the inspected plant. As a second step, proper sampling point selection and subsequent sampling and analysis may follow after the inspection team had developed a sufficient understanding of the plant. In order to be able to do so, relevant information should be provided by the plant operator to the inspection team.

Thus, the following steps were considered necessary:
1. Prompt analysis of the following types of samples immediately after commencement of inspection activities:

- air samples taken from the plant environment (close to reaction vessels, in- and out-lets, etc.) and around the plant;
- soil, dust, and ground samples taken as above;
- samples taken from the waste water channel, and the reactor ventilation system;
- wipe samples taken from surfaces in the plant area.

2. Sampling and analysis at measurement points selected after careful design evaluation of the plant, such as:

- joints most likely to retain traces of previously produced chemicals;
- valves and/or outlets;
- elements inside the waste treatment station/channel;
- part of the reactor ventilation system/gaseous effluent treatment system;
- structural elements in the factory area which are likely to retain residues of chemicals present in the plant environment (validation of these points by analytical methods).

Some of these measurement points may already have been selected under 1 and will not be sampled a second time. However, it is considered of crucial importance that the inspectors develop a certain understanding of the plant design in order to sample at valid points. Thus, it will be necessary that the inspection team be given a plant design map together with a description of the technology used and be allowed to verify the correctness of that information by visual checks.

3. As a crucial additional effort, the team has also to verify whether clean-outs at the site can be ruled out.

Indicators to be looked for in this context may be, for example:

- visible signs at joints and other parts of equipments indicating recently performed replacements of equipments, renewed joint packings, etc.;
- signs for recent extensive cleaning operations at the site, to be detected by visual inspection;
- residues of detoxification operations (e.g., decontaminants at surfaces of reaction vessels, pipelines and other equipment, in ground and building structures, and in the interior of the equipment).

As another inspection method applicable in this phase as in fact in all phases, the inspectors may conduct interviews with facility personnel.

In case that residues of schedule-1-chemicals were detected at an inspected site (most probably at trace level), the inspection should immediately proceed to phase three (resolution of anomalies). The same applies in case that unmistakable signs for extensive clean-outs are encountered which might be residues of a covering-up. It should be underlined here that these anomalies are so far merely verification signals yet no proof of a violation.

In case that no such verification signals were encountered, a challenge inspection should proceed to phase two.

**2. Phase two**

The aim in this inspection phase is to assess whether or not the plant poses an immediate and high risk to the objectives of the convention. In this context, risk is understood to comprise both the potential CW capability of the plant and its roughly estimated capacity, in case it was assessed as being capable. CW capability is defined for the purpose of this discussion as being made up of the following increments: "chemical capability" (i.e., the availability of requisite chemicals), "technological capability" (i.e., the availability of requisite equipment), safety features, and the overall security regime at the inspected site.

The purpose of this particular phase is to provide for a sound decision by the team as to whether or not the results of phase one, if all negative (i.e., no detection so far of schedule-1-chemicals or signals for potential cover-up attempts), would suffice for the implementation of the inspection mandate.

This phase may become necessary because for a challenge inspection to be able to conclusively demonstrate compliance or prove violation, an inspection team will have to take into account the possibility that a violator had developed counter-inspection strategies which would

to some extent obstruct the team's ability to detect a violation.

To assess whether or not an inspected plant poses an immediate and high risk to the objectives of the convention, it was considered inappropriate to request a plain statement from the facility management concerning the capability of the plant to produce schedule-1-chemicals. Rather, the approach suggested is to request a number of statements and documentations, subject to verification, which would allow the inspection team to carry out an independent if qualitative risk assessment.

The answers to be provided by the plant management will have to be backed-up by maps, the plant design map already submitted in phase 1, and will be validated by independent verification activities such as visual checks and analytical tests of chemical storage (preferably head-space analysis). Again, an increment of this phase would be to assure that no signs of masking activities are detectable.

These statements and documents might include:

- The territorial situation of the site in relation to urban areas (cities, villages, etc.);
- A statement on special preparation at the plant for providing safety against chemical accidents (such as arrays of special chemical sensors or alarms, installations suitable for rapid decontamination of the site, availability of specific decontaminants at the plant, availability of individual protection equipments and its nature, etc.);
- A statement on special equipment installed at the plant, such as reaction vessel with inner surface coatings inert against highly corrosive gases (such as HF), or measures taken to provide for leak-protection to an unusual extent;
- A statement on the utilization (presence at the factory) of chemicals to assess the plant's "chemical capability", and notably of chemicals listed under schedules 2 and 3 of the convention.

For the evaluation of the risk that the plant poses to the objectives of the convention, the following assessments were conceived in combination:

1. A qualitative downwind hazard evaluation for nearby urban areas assuming production of a schedule-1 warfare agent at the factory, based on plant size and actual territorial features of the area.

2. Assessment of the plant's capability to produce a schedule-1-chemical based on available **chemicals** (starting materials, intermediates, final products).

3. Assessment of the plant's capability to produce a schedule-1-chemical based on the existing **technological**

**and safety lay-out** (including equipment present, systems providing for plant safety such as monitoring of chemicals at the site, preparations for large-scale decontamination, and waste and effluent treatment, relationship to other plants if the inspected facility is part of a larger complex, etc.).

4. Assessment of the security regime at the site.

Here is an example for how such assessments may be carried out: In the GDR trial challenge inspection, a computer program for the generation of synthesis paths was used for evaluating the "chemical capability". For illustrative purposes, some general information on the program follows:

The program used is called RDSS (Reaction Design by Synthone Substitution) and has been jointly developed by the Chemiekombinat Bitterfeld, and the Central Institute for Cybernetics and Information, GDR Academy of Sciences, Berlin, with the aim to compute synthesis strategies or break-down paths of chemicals. It may be applied to compute potential synthesis paths from chemicals present at a site into chemicals listed under schedule 1.

The program uses structural features of the parent chemicals in form of synthones and a database compiling chemical reactions published during the past 10 or so years as reported by the Chemical Abstracts Service. Synthones form the "knowledge-basis" of this data-base. Starting from the structural information of a parent chemical, generation of son chemicals is thus possible, and vice versa. It is also possible to combine a parent chemical with a remote son chemical (superposition of son chemical generation with break-down computation) in order to identify possible synthetic paths between the two.

The program uses eight different levels of discrimination for excluding chemically improbable reactions. It is planned to further study the potential application of this program for verification purposes.

In a more general way, approaches and concepts which have been developed for the conduct of risk assessments in other fields may to some extent be applicable here.

5. The overall risk assessment would be the result of all these partial assessments in combination. In case that the inspection team encounters features at the inspected site which, in the judgment of the team and based on best international practice at industrial plants of the type inspected, may point to an immediate and significant risk, the inspection could enter into phase 3. Otherwise, the team could conclude inspection activities.

## 4. Phase three

Three types of plants may reach inspection phase three:

Plants showing verification anomalies in the first two inspection phases, such as
- detected residues of prohibited chemicals;
- detected traces of plant modification prior to the inspection;

and plants being assessed by the inspection team as immediate and high risk to the objectives of the convention.

In the first case, i.e. in case that prohibited chemicals were in fact detected (most probably at trace level), the following steps may be considered for phase-3 inspection activities:

- sampling for off-site confirmation analysis, preferably using mass spectrometry (it is assumed that a positive on-site analysis would be backed up by a double and a blank test), for samples taken at the same and additional sampling points;
- the plant management and/or the representative of the national authority of the inspected state should be requested to provide an explanation (which should be recorded in the inspection report). To the extent possible, data in that statement should be verified as well.

In case that the inspection team considers the provided explanation insufficient, ambiguous, or otherwise unsatisfactory, and hence serious doubts arise as to whether or not a violation might in fact have taken place, the inspection team would decide to proceed to activities under phase four.

The second type of plant to consider is one where alterations were detected. Such alterations might have been:

- clean-out activities including decontamination;
- physical replacements of equipments in place.

For a typical industrial chemical plant, it seems reasonable to assume that an inspection team may face one of the following situations which would generate signals for the inspectors:

- plant under routine overhaul (i.e., non-producing)
- repair operations after accidents etc.
- routine clean-out to prepare for new production
- recently started new production cycle
- other routine maintenance operations

Given the time requirements of challenge inspections, it certainly will be possible to discriminate such

legitimate operations against extensive express cover-up attempts such as external and internal plant decontamination, replacement of a number of sensitive equipments, breaking and re-fitting of joints in a rather large number, etc.

In order to resolve anomalies, the nature and timing of alterations ought to be assessed by the team. For doing so, a facility statement should be requested explaining the time (whether the alteration was done before or after information on the imminence of an inspection), nature, and purpose of the alteration. On this basis, the inspection team should attempt to

- assess the plausibility of the given statement;
- verify, to the extent possible, details given in this statement (e.g., by record evaluation, interviews, sampling and analysis of decontaminants used, visual inspection, etc.), the level of intrusion being governed by the nature and extent of the alteration detected;
- evaluate the potential consequences in case of major alterations and assuming that the statement, to the extent not verifiable any more, may have been falsified deliberately.

Based on these activities, the team may find itself in a position either to conclude that despite detected alterations no indications of an actual violation or a severe risk to that end were encountered, or that such indications exist. In the latter case, the team would proceed to inspection phase four.

The third type of plant to consider is one where no traces of schedule-1-chemicals were detected and no signs of cover-up activities were encountered either while the team still concludes that the plant, given its access to raw material and its technological lay-out and security and other features, may pose an immediate and significant risk to the objectives of the convention. The baseline for such a judgment should be the best international practice at chemical industry plants of a comparable type. Such a plant would consequently show a number of unexpected features as compared to other, and notably state-of-the-art plants.

At that stage, an inspection team might decide to do one of the following:

- assess the plausibility of the plant design and of other features encountered;
- carry out additional analytical investigations, as a back-up for the already performed analytical tests and at other sampling points;
- verify facility records, operating records, and further documentation for confirmation of the facility statement.

The inspection would proceed further into phase four only in cases where evidence casts doubt on the compliance at the plant.

## 5. Phase four

Plants reaching this inspection phase are highly suspicious of actually having violated the convention because of the verification results so far encountered, which may be one or more of the following:

- confirmed identification of schedule-1-chemicals in the facility plus inappropriate (in the judgment of the inspection team) explanation provided by the plant operator and/or the representative of the national authority for the presence of these chemicals;
- unmistakable signs for most recent cleaning or replacement operations which appear to depart from the normal pattern of such operations at industrial plants;
- appearance of a number of features which let the inspection team conclude that the plant may have been designed in such a way as to be capable of producing schedule-1-chemicals on a significant scale while obstructing verification effectiveness to some extent.

The inspection has to exploit all possibilities to conclusively prove that a violation has in fact occurred, or that despite verification signals encountered so far a violation can be ruled out. The methods applied by the inspection team and the level of intrusion will have to be such as to enable the team to arrive at conclusive inspection results as long as the facility operator and the representative of the inspected state continue to co-operate.

Assuming continued such co-operation (which itself can be regarded as a signal in the verification process indicating though not demonstrating compliance), some of the following steps might be taken by the inspection team depending on the specific situation:

- Detailed auditing of plant records, additional documentations (e.g., bills and contracts), etc.;
- Interviews with plant personnel, medical check-ups to identify signs of earlier intoxication and/or exposure to prohibited chemicals;
- Verification of documents about the history of the plant (accident protocols, reports of national or international inspections/visits in frame- works other than verification of the chemical weapons convention, such as commercial contacts, environmental inspections, etc.;
- Verification of related facilities/plants if the inspected plant is part of a larger complex;
- Submission of data to balance the material consumed for legitimate production at the site with subsequent verification of these data to the extent still possible

(in particular, cross- examination with data from outside the facility, if available at the Technical Secretariat);
- Further analytical investigations in and around the plant in order to find traces of prohibited chemicals or decontamination agents.

Other intrusive inspection methods may be applied as well depending on the circumstances.

**Summary**

The inspection methodology described in the present working paper for the conduct of challenge inspection in industrial plants has been partly tested in the GDR trial challenge inspection at the chemical combine in Bitterfeld. During this exercise and in the evaluation of the results of the trial, the participants concluded that such an approach might

- facilitate the implementation of the principle to apply the least intrusive verification methods possible;
- provide guidance for future inspectors for how to implement the inspection mandate;
- allow the inspection team to appropriately and effectively respond to potential counter-inspection strategies;
- allow, for the cases of such an inspection at innocent plants, to keep the level of intrusion modest while still retaining the ability to detect signs of violations had these occurred.

Preliminary experience in applying the concept described in this working paper seems to suggest that such a methodological approach will help to keep the number of challenge inspections with inconclusive results very low.

It stands to reason that all procedures suggested should be applied in a very flexible manner and that the inspectors should have the necessary skills, knowledge and experience.

CONFERENCE ON DISARMAMENT

CD/998
CD/CW/WP.294
12 June 1990

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GERMAN DEMOCRATIC REPUBLIC

# Application of Trace Analysis to Exploit Memory Effects in Challenge Inspections

## Introduction

For an effective conduct of challenge inspections, appropriate analytical concepts, methods, and instruments are essential. They will have to combine high specificity and sensitivity with as little intrusiveness as possible. Also, methods used should have a certain capability to provide information about previous activities at the inspected site. Other important features would be portability and reasonable cost.

This working paper briefly describes an approach developed in the GDR's trial challenge inspection in an chemical industry plant (see also the corresponding report which has been submitted as a separate working paper). An attempt was made in this trial to develop sampling and analytical methods for quick on-site verification using a portable ion mobility spectrometer, in order to exploit memory effects of a chemical plant. Investigations are still being conducted to further improve the concept.

## 1. Development of methods in pre-inspection laboratory experiments

### 1.1. General Information

Instrument used: prototype transportable PC-supported IMS, developed at the Central Institute of Isotope and Radiation Research, GDR Academy of Sciences, Leipzig

### Instrument Calibration

In order to predict the position of nerve agents on the flow-time axis, the ion mass - ion mobility - dependence was established using several nerve agent simulants. The concentrations were in the range of 5 to 50 ppb using a gas permeation chamber. Dimerization peaks were usually recorded at concentrations of 5 to 35 ppb, and the peaks corresponding to ions of the type $M_2H^+$ (M = molecule of the substance to be determined) - the energetically most stable ions generated - were usually selected for identification purposes. Also, peaks corresponding to

ions of the type $M(H_2O)_6H^+$ were used for that purpose. The corresponding relationships were:

- for ion type $M_2H^+$:

$$\lg m = -0.544 \times K_o^+ + 3.176 \quad (r = 0.998)$$

- for ion type $M(H_2O)_6H^+$ and ion type $M_2H^+$:

$$\lg m = -0.510 \times K_o^+ + 3.137 \quad (r = 0.995)$$

These correlations are consistent with data published in literature.

In all further experiments, DIMP (diisopropyl-methylphosphonate) was used as the model substance simulating schedule-1-chemicals.

### 1.2. Pre-inspection laboratory experiments

A number of experiments was conducted before the trial inspection in order to develop methods which would allow to:

- validate sampling points selected in a plant;
- identify traces of organophosphorous schedule-1-chemicals against the background of chemicals present;
- detect traces of chemicals deposited in the plant area by production activities which had occurred before commencement of the inspection.

#### 1.2.1. Detection of DIMP in mixtures containing an excess concentration of organophosphorous pesticides

The following organophosphorous pesticides (in form of formulations and/or pure compounds) were used in the experiments:

- Bi-58 EC (pesticide formulation containing dimethoat)
- Wofatox (pesticide formulation containing parathion-methyl)
- Fekama Tribuphon EC 50 (pesticide formulation containing butonate)
- DDVP (pure chemical)
- methamidophos (pure chemical)

DIMP detection and identification was performed from gas samples as well as in the head space of liquid mixtures (placed inside a diffusion chamber). The following conclusions were drawn:

- At a DIMP concentration in the gas phase of approximately 0.04 mg/m³ and a DIMP to pesticide ratio of 1 : (13 500 ... 22 000) for pesticide formulations, which corresponds to a ratio of 1 : (1 000 ... 10 000) for the pesticide itself, it was in all cases possible to clearly identify the $(DIMP)_2$ peaks. These data were further

substantiated in experiments using pesticides as pure chemicals.
- In liquid mixtures of DIMP in pesticide formulations (pesticide concentration at a thousand-fold excess and above), it was possible to clearly identify DIMP from head space samples of these mixtures several hours after preparation of the mixtures.

Examples of the IMS spectra generated in these experiments are presented in figures 1 to 4.

**1.2.2. DIMP identification from rubber material**

In order to investigate the memory effect to be expected by absorption of chemicals in organic material used for joint packings etc., a piece of rubber tube was inserted into a parathion-methyl formulation containing approximately 0.1 per cent DIMP, for 16 hours. The sample was wiped off and placed under a laboratory hood. For measurement, it was placed into the diffusion chamber connected to the IMS and replaced under the hood again after measurement. Signal recording was conducted at 120 hours, 240 hours, 410 hours, and 580 hours after initial sample preparation.

As can be seen from figure 5, even 580 hours after the initial sample preparation identification of DIMP remained possible with sufficient reliability.

**1.2.3. Development of wipe tests**

In order to evaluate the detection limits for wipe tests on metal surfaces potentially contaminated with schedule-1-chemicals, between 10 and 100 microliters of a solution containing 10 micrograms per ml DIMP in n-hexane were transferred onto aerosol filter paper (diameter 2 cm). The samples were air-dried for 2 minutes and placed into the IMS sampling chamber for subsequent analysis. The detection limit thus established was approximately 1 microgram DIMP. The test was then repeated with a DIMP - pesticide mixture (parathion-methyl formulation). This test confirmed the detection limit.

In a subsequent experiment, a steal surface was contaminated with the DIMP-pesticide mixture (1 microgram DIMP was thus applied onto a surface of approximately 5 square centimeters). After 2 minutes, the steal surface was wiped off with filter paper which was then placed into the IMS inlet chamber. The signals recorded did confirm the detection limit estimated in the first experiment.

For illustration, the recorded spectra are presented in figures 6 and 7.

## 2. Trial inspection analytical results

Details on the conduct of the trial inspection and the basic methodology applied are reported in two other working documents, and will not be repeated here. Only the analytical data are set out in the following section.

### 2.1. Selection of measurement points

The measurement points selected after plant design analysis and visual inspection of the plant are illustrated in figure 8, in a simplified way. The following points were selected:

1. Ambient air samples taken outside the factory building using an air filtration system connected to the instrument.

Justification: Rapid environmental analysis, establishment of the analytical baseline for other tests.

2. As under 1, without air filtration.

Justification: Rapid environmental analysis, detection of prohibited chemicals and actual production.

3. Air samples taken inside the factory building.

Justification: Detection of prohibited chemicals as well as pesticides produced in the plant, indication of air tightness of the equipment installed.

4. Air samples taken very close to the inner wall surface of the building.

Justification: Exploitation of memory effects for the detection of previous activities.

5. Sniff-test from joints connecting to reaction vessels

Justification: As under 4

6. Wipe-samples taken from reactor surfaces

Justification: Detection of residue concentrations of chemicals at levels to be expected after cleaning or decontamination of surfaces, detection of residues of decontaminants.

7. Dust sample taken from the ground inside the factory building

Justification: As under 4

8. Samples taken from the interior of reaction vessels

Justification: Detection of traces of former production in case of a recent clean-out and re-start of innocent production.

**2.2. Results at measurement points 1 to 4**

These results are summarized in figure 9.

While in the atmosphere outside the plant building, only chloro benzene could be detected, parathion-methyl could be clearly identified in the interior plant atmosphere. The detected pesticide concentrations right above the wall surface were considerably higher than approximately one meter away from the reaction vessels.

Conclusions drawn:

1. The equipment at the site was clearly unsuited for nerve gas production in terms of gas-tightness. Air in the interior of the factory was contaminated with the product of the plant and with chemicals used as starting materials.

2. The higher concentration of parathion-methyl at the wall was interpreted as a memory effect. Chemicals present at the site will stay for prolonged periods of time in these structures and will remain detectable with trace analytical methods.

**2.3. Measurements at point 5**

The results of measurements of samples taken from joint packings are illustrated in figure 10. Air was sucked-off from a joint slit. In a validation experiment, approximately 45 ppb DIMP were added by insertion of a diffusion tube into the gas stream. DIMP was clearly identifiable against a huge excess concentration of chemicals absorbed by the material and released during sampling. This parathion-methyl concentration in turn demonstrated the feasibility of exploiting memory effects in such structures.

As a back-up to these results, laboratory investigations were conducted with the types of packing material actually used in the factory. The test conditions were those described in para 1.2.2. The results are presented in figures 11 and 12. The simulant remained detectable for more than 235 hours from both materials tested. Preliminary laboratory results suggest a much longer residence time of chemicals adsorbed or absorbed by packing materials. Additional tests are still under way.

Based on the experimental data so far available, it seems feasible to combine trace analytical methods for the identification of certain chemicals with visual checks to detect indications of joint packing replacements. Such an

approach should have a considerable retrospective capability while also enabling to identify chemicals which were previously used or produced in the plant even without sampling from the interior of the vessels etc. (i.e., in as non-intrusive way which would not interfere with normal facility operations).

**2.4. Wipe test results (measurement point 6)**

A solution of DIMP in n-hexane was sprayed onto the surface of a reaction vessel 2.5 hours before the test. The amount so applied to the steel surface was approximately 10 microgram per square centimeter. The sampling device was an aerosol filter paper (diameter 2 cm). After sampling, the filter paper was inserted into the inlet head of the IMS. The results so recorded are presented in figure 13. DIMP was clearly identifiable in the sample.

Additional tests are still under way to establish the retrospective time coverage capability of that test.

**2.5. Dust sample results**

Dust samples taken from the factory ground were analyzed. In a validation experiment, such dust samples were transferred into the laboratory and spiked with different concentrations of DIMP. Several hours after preparation, the samples were placed into a special inlet system of the IMS and spectra were subsequently recorded. The results are presented in figure 14. DIMP can clearly be identified at 10 and 100 micrograms in a 7 grams dust sample. Additional tests are still under way.

**2.6. Identification of DIMP in the actual parathion-methyl product solution (sampling point 8)**

A sample was taken from a reaction vessel containing the final product solution, and taken to the laboratory. DIMP was added at a concentration of 0.04 per cent (in relation to parathion-methyl). It was assumed that this concentration might represent a residue concentration of a former production after cleaning the plant and re-starting synthesis of another product, in the first batch.

The time dependence of the recorded signal is presented in figure 15. This time dependence may be a result of alterations in the sample over time, partial phase separation, and evaporation. However, the results clearly show that traces of the simulant remain detectable at the concentration chosen.

## 3. Results and conclusions

1. The combination of sampling techniques and analytical instrumentation used in the GDR trial challenge inspection was demonstrated to be applicable, in principle, for:

- the identification and/or validation of relevant sampling points, in particular those where enrichment of chemicals formerly having been deposited in plant structures has occurred, and
- the identification of a nerve agent simulant against an excess concentration of substances which are chemically very close (notably, organophosphorous pesticides).

2. In preliminary investigations, analytical concepts were developed and successfully tested which allow to exploit memory effects for retrospective detection and identification of production in a chemical plant. The investigations are being continued and a follow-up report will be presented at a later stage.

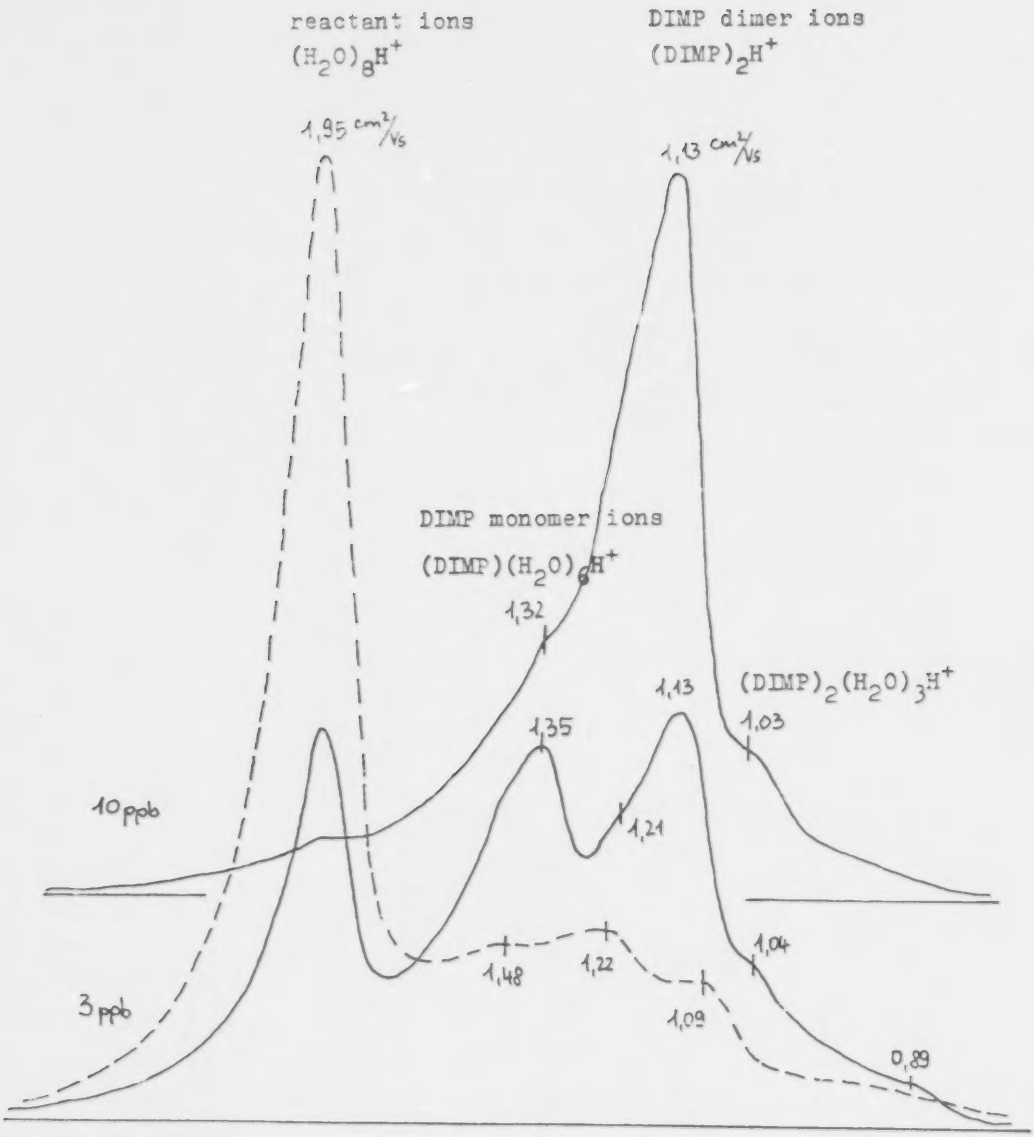



Fig. 1: IMS spectra recorded for 3 and 10 ppb DIMP in air

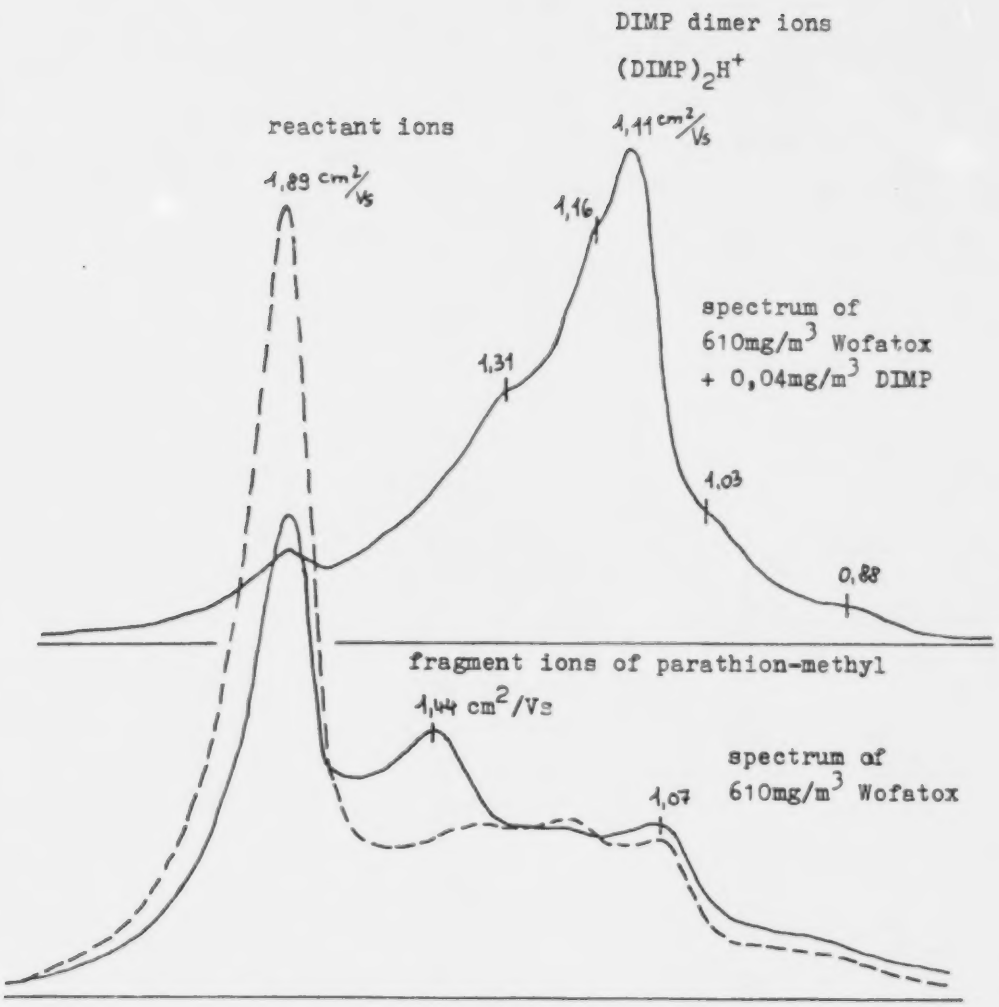



Fig. 2: Influence of 610mg/m$^3$ Wofatox on the shape of the spectrum of 0,04mg/m$^3$ DIMP

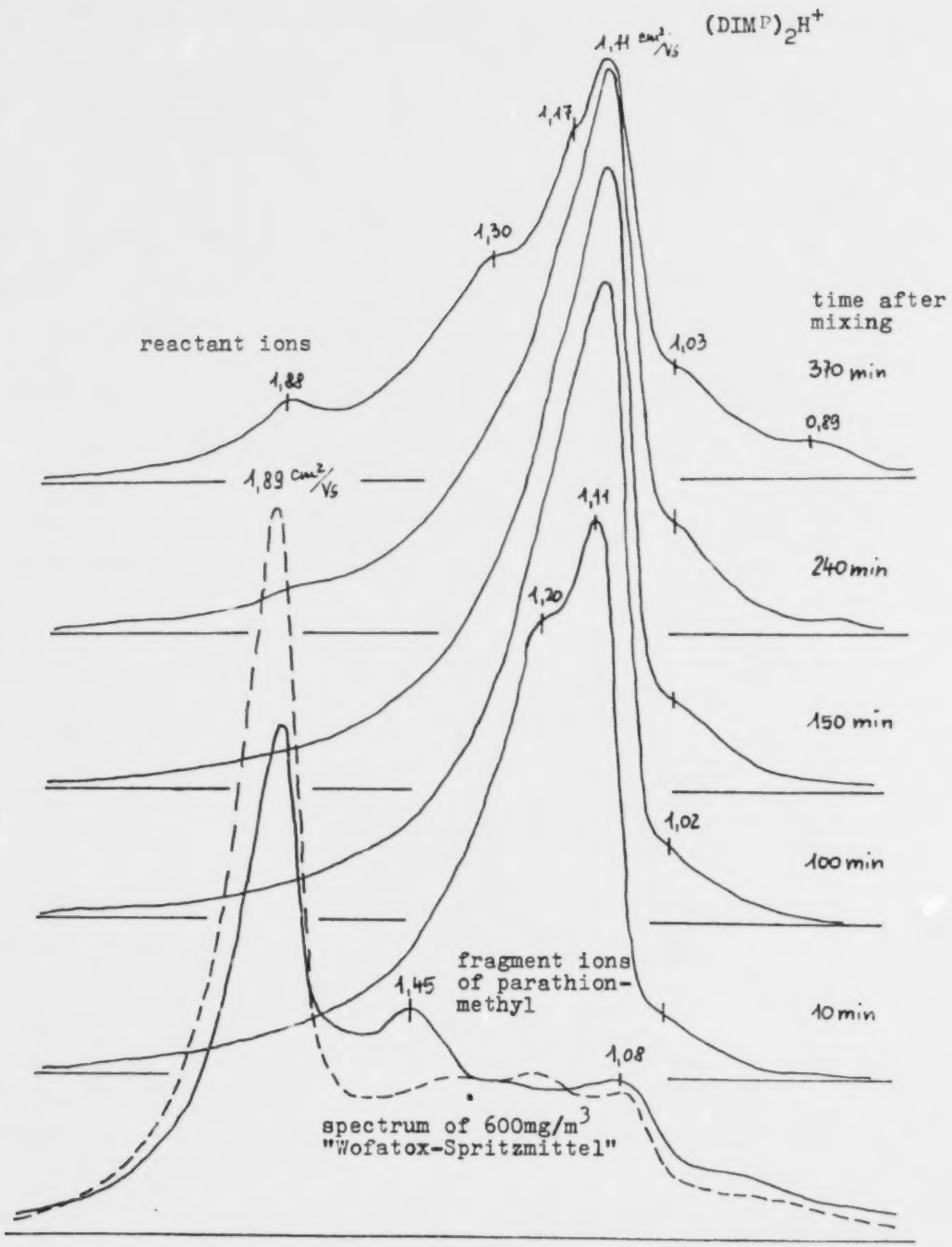



Fig. 3: Spectrum of a mixture of "Wofatox-Spritzmittel" and $1\,^{o}/_{oo}$ DIMP

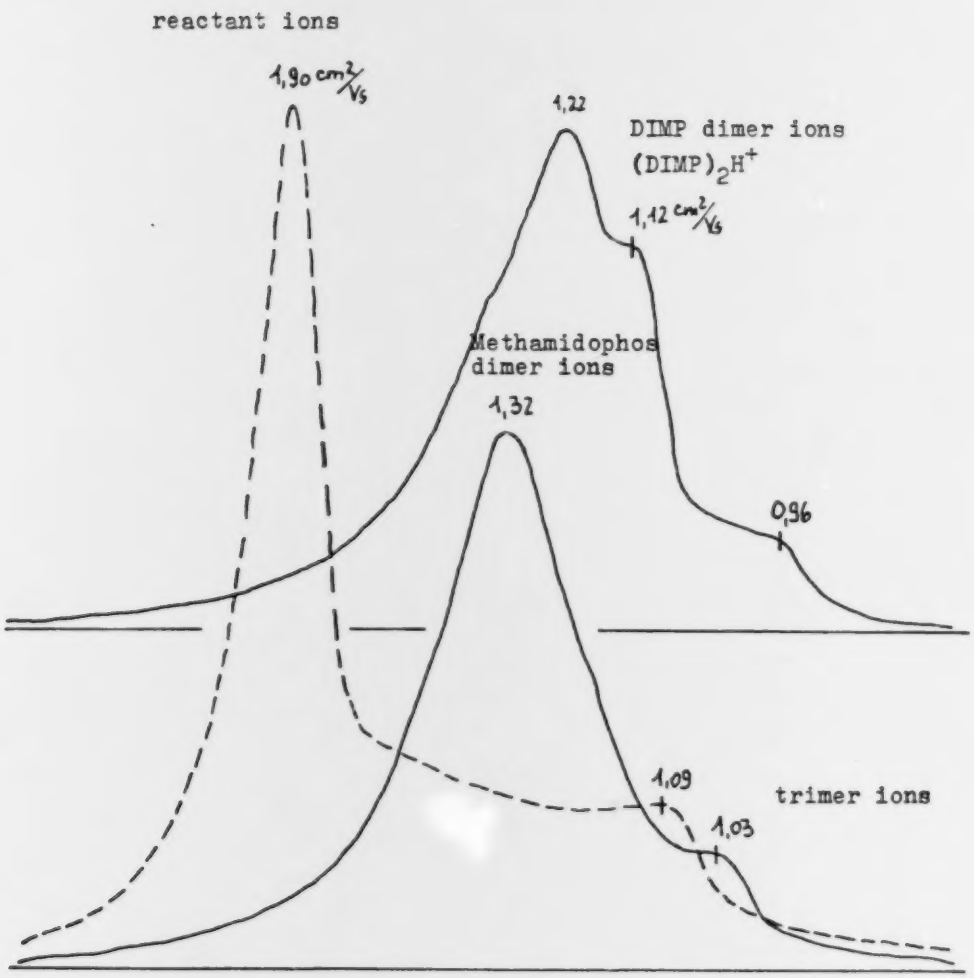



Fig. 4: Spectrum of a gasphase mixture of
10 ... 20 ppm "Methamidophos" and 5 ppb DIMP

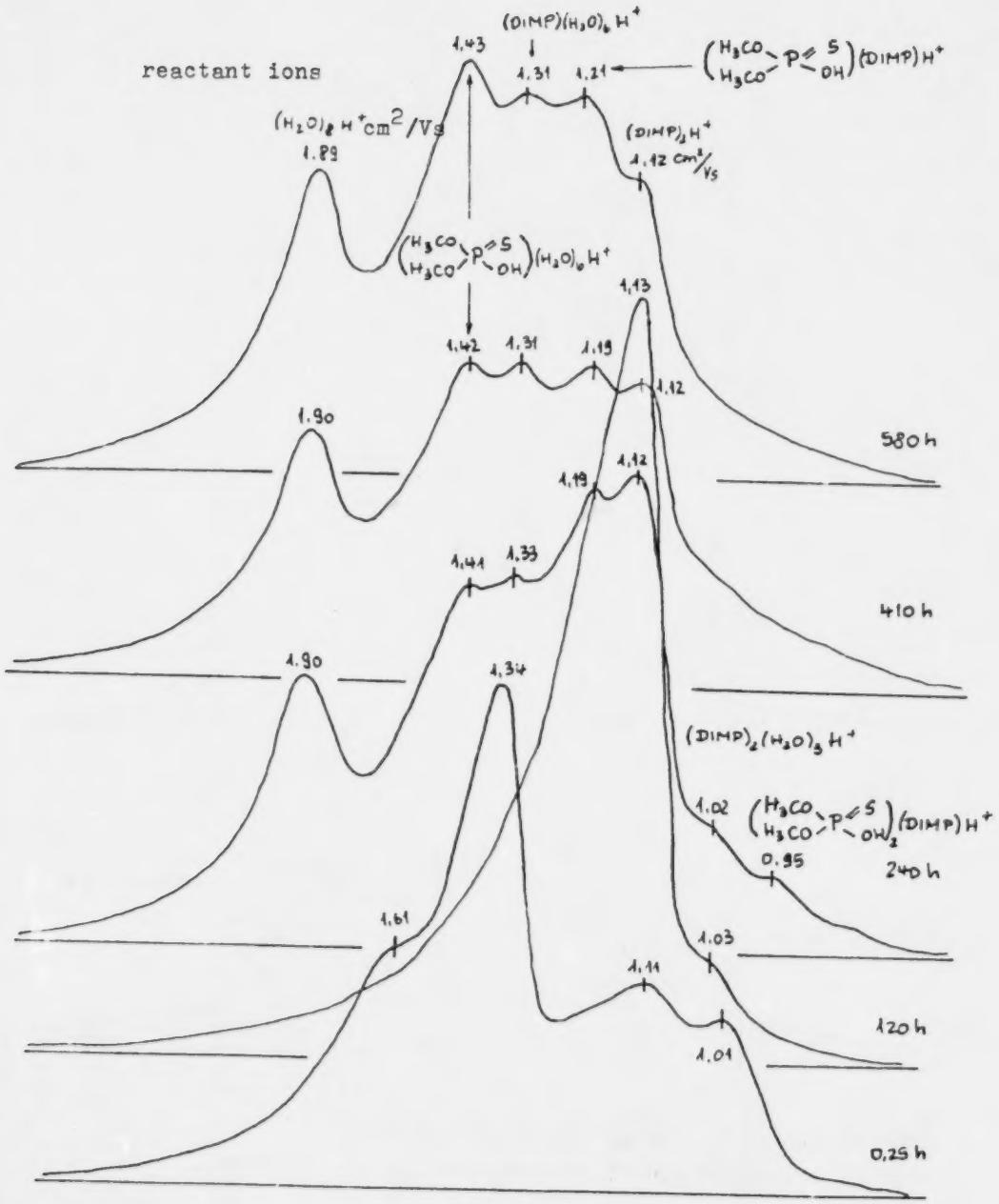



Fig. 5: Spectra recorded from a rubber sample contaminated with Wofatox + $1^{o}/_{oo}$ DIMP mixture, with increasing storage time

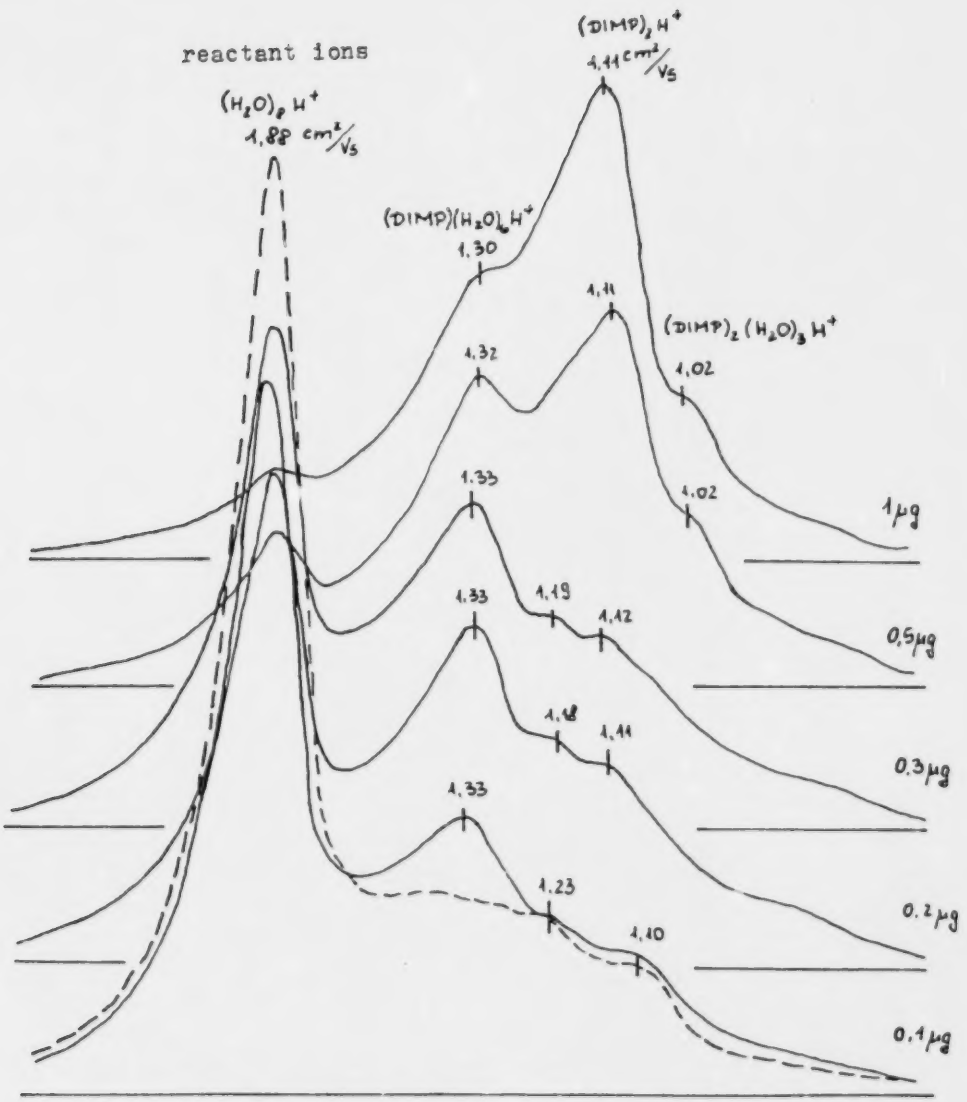



Fig. 6: Spectra of DIMP applied on aerosol filter paper, for different contents of DIMP

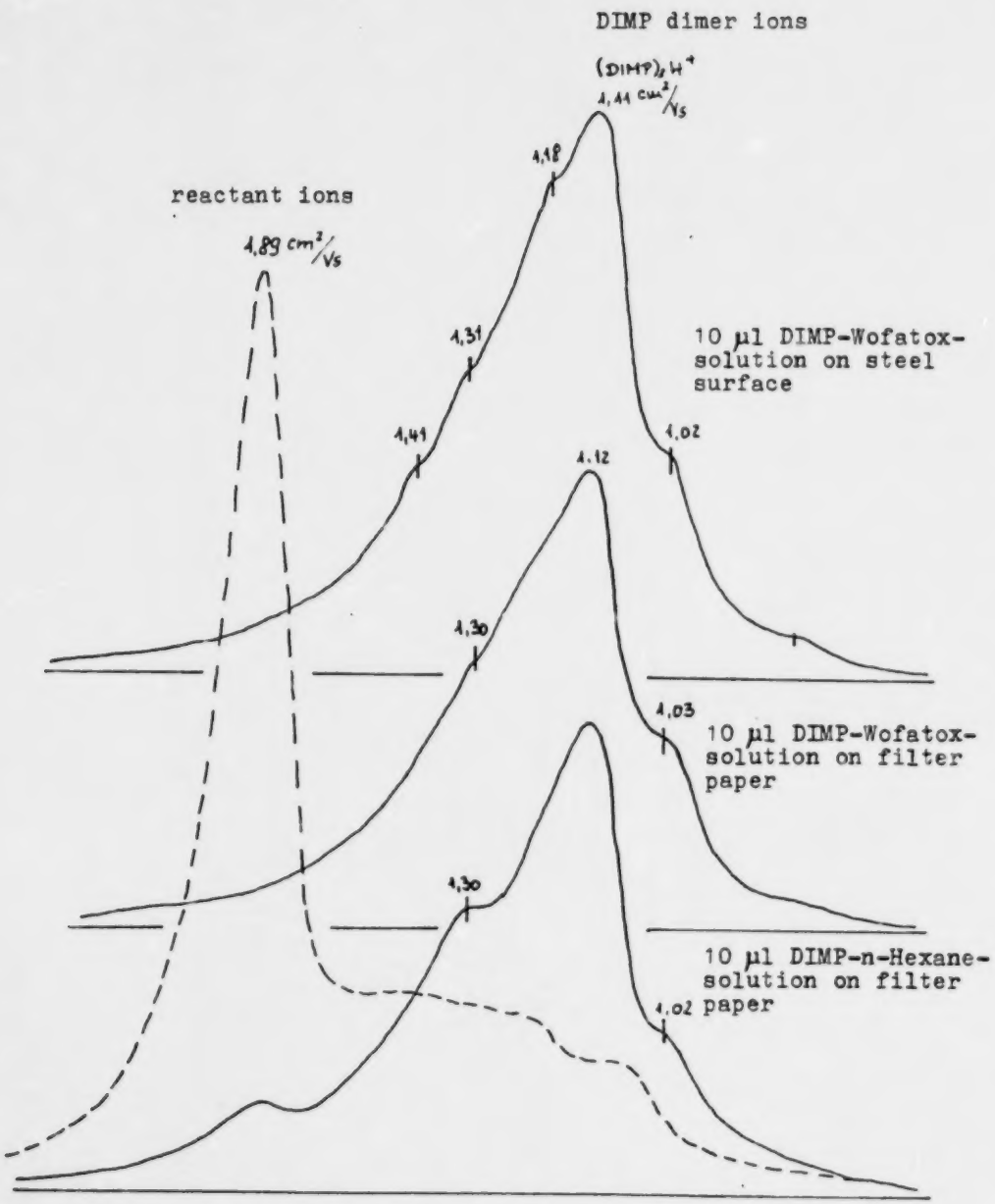
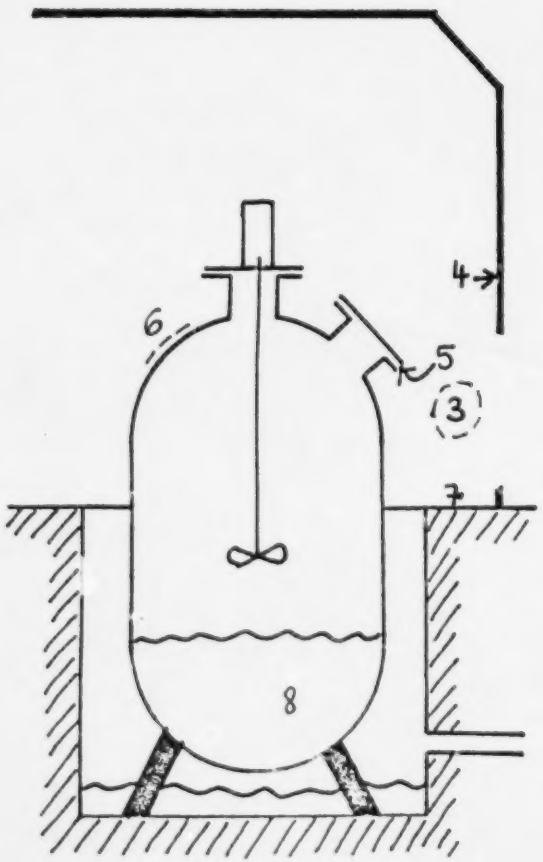



Fig. 7: Simulation of wipe-tests on a steel surface contaminated with Wofatox-DIMP-solution of a concentration of 0,1 mg DIMP / ml solution, the DIMP content was about 1 µg in all tests




1
2

**Measurement points selected**

1 Air sample taken outdoors, filtered through activated carbon
2 Air sample taken outdoors, not filtered
3 Ambient air sample taken indoors
4 Air sample taken from a wall surface
5 Sniff-test from joint packing
6 Wipe test on reactor surface
7 Dust sample from ground or wall
8 Reactor fill sample

Fig. 8: Measurement points selected for trace analysis at the Wofatox - plant (schematic)

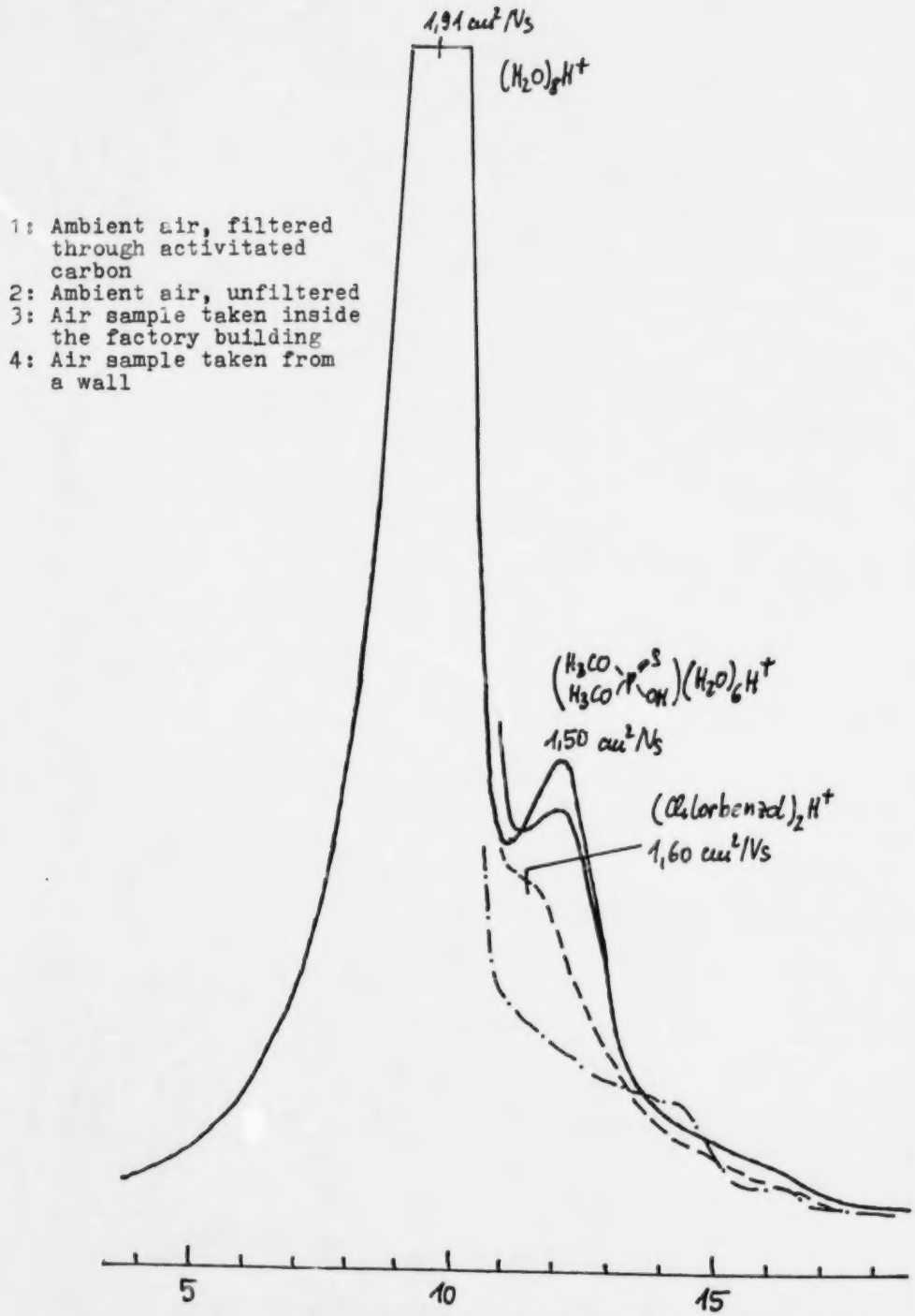



Fig. 9: IMS signals recorded at measurement points 1 to 4

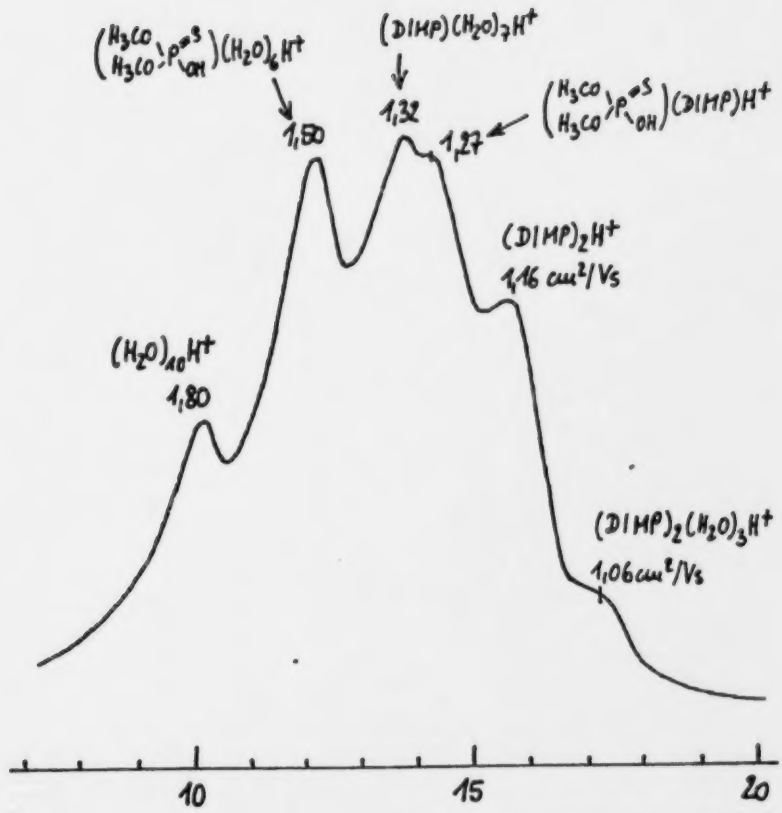



Fig. 10: IMS spectrum recorded at measurement point 5, approx. 45 ppb DIMP were added into the gas stream sampled from the joint

Fig. 11: IMS spectra recorded from fitting material (Kautasit), which was submerged into a parathion-methyl DIMP - mixture ($1^{o}/_{oo}$) for 16 hours, time dependence

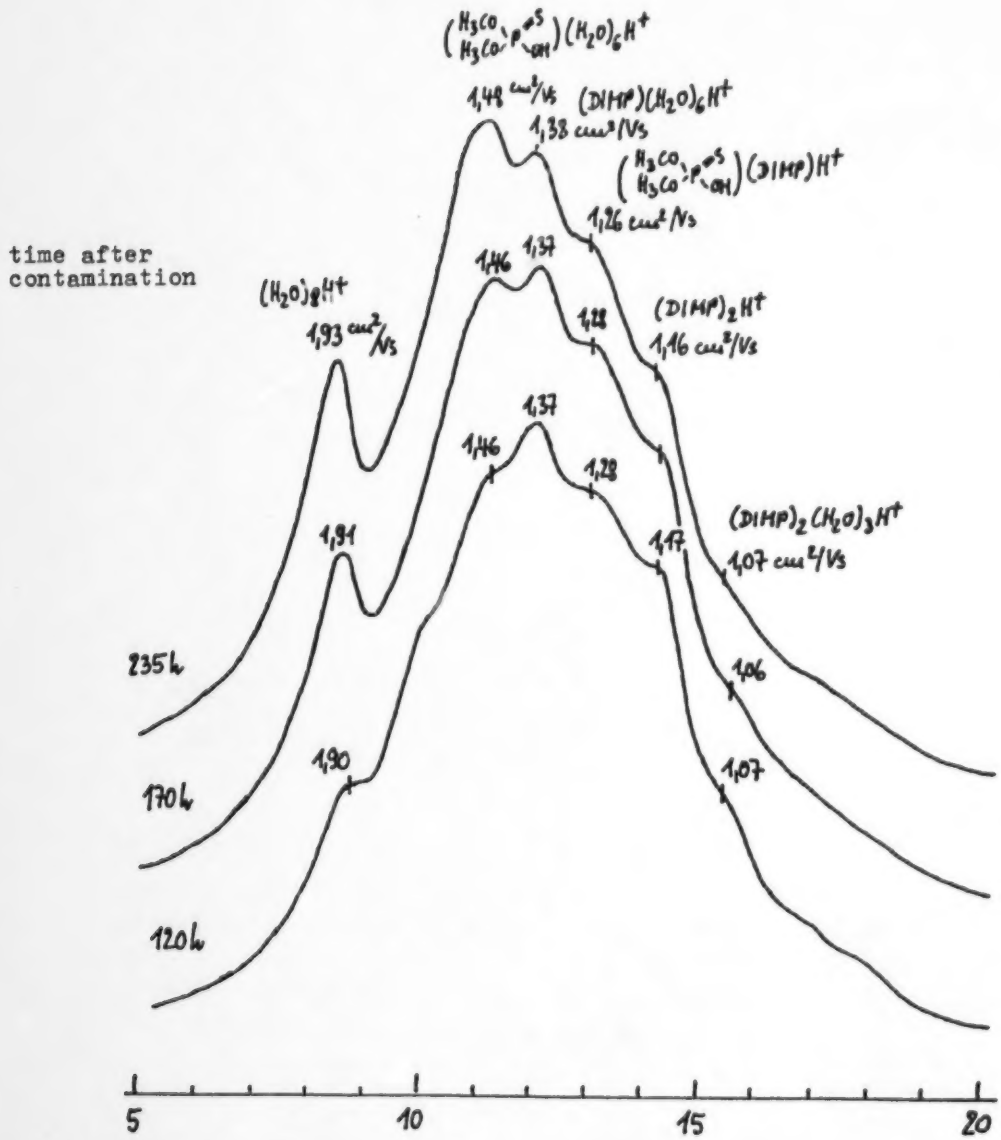
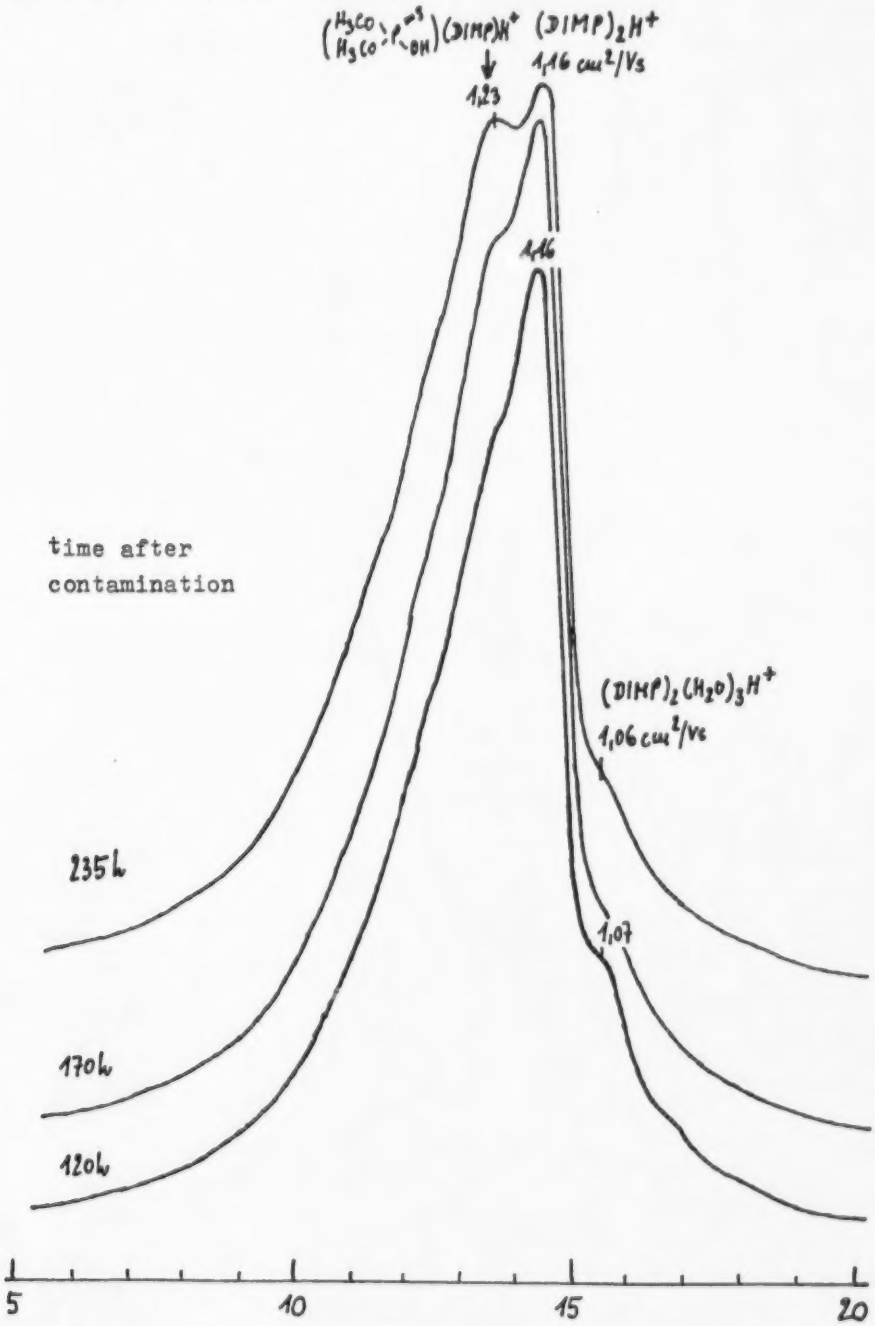


Fig. 12: IMS spectra recorded from packing material (PTFE) which was submerged into a parathion-methyl - DIMP mixture (1 $^{o}/_{oo}$) for 16 hours, time dependence



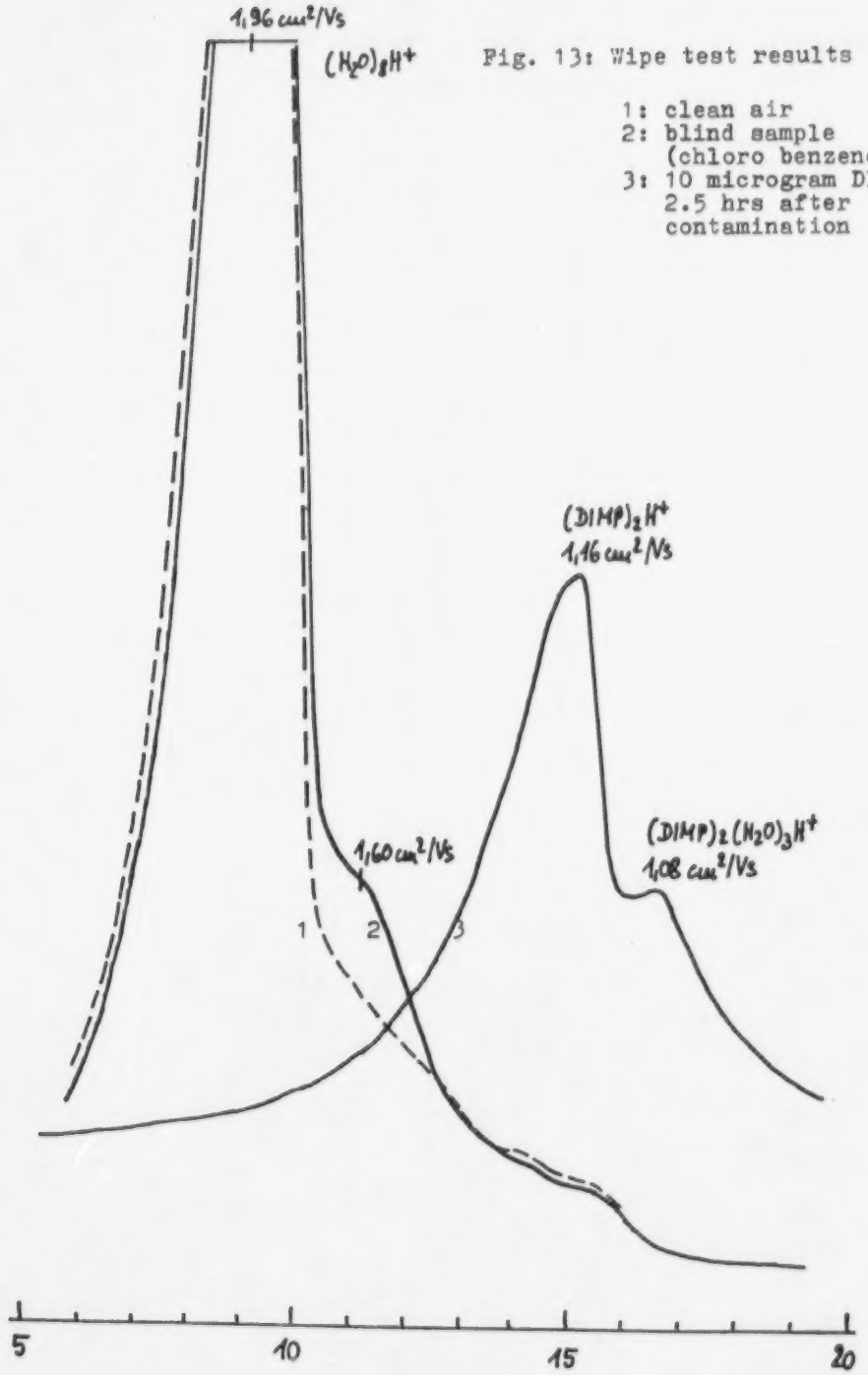



Fig. 13: Wipe test results

1: clean air
2: blind sample
(chloro benzene)
3: 10 microgram DIMP
2.5 hrs after
contamination

Fig. 14: IMS signals recorded from dust samples contaminated with different concentrations of DIMP

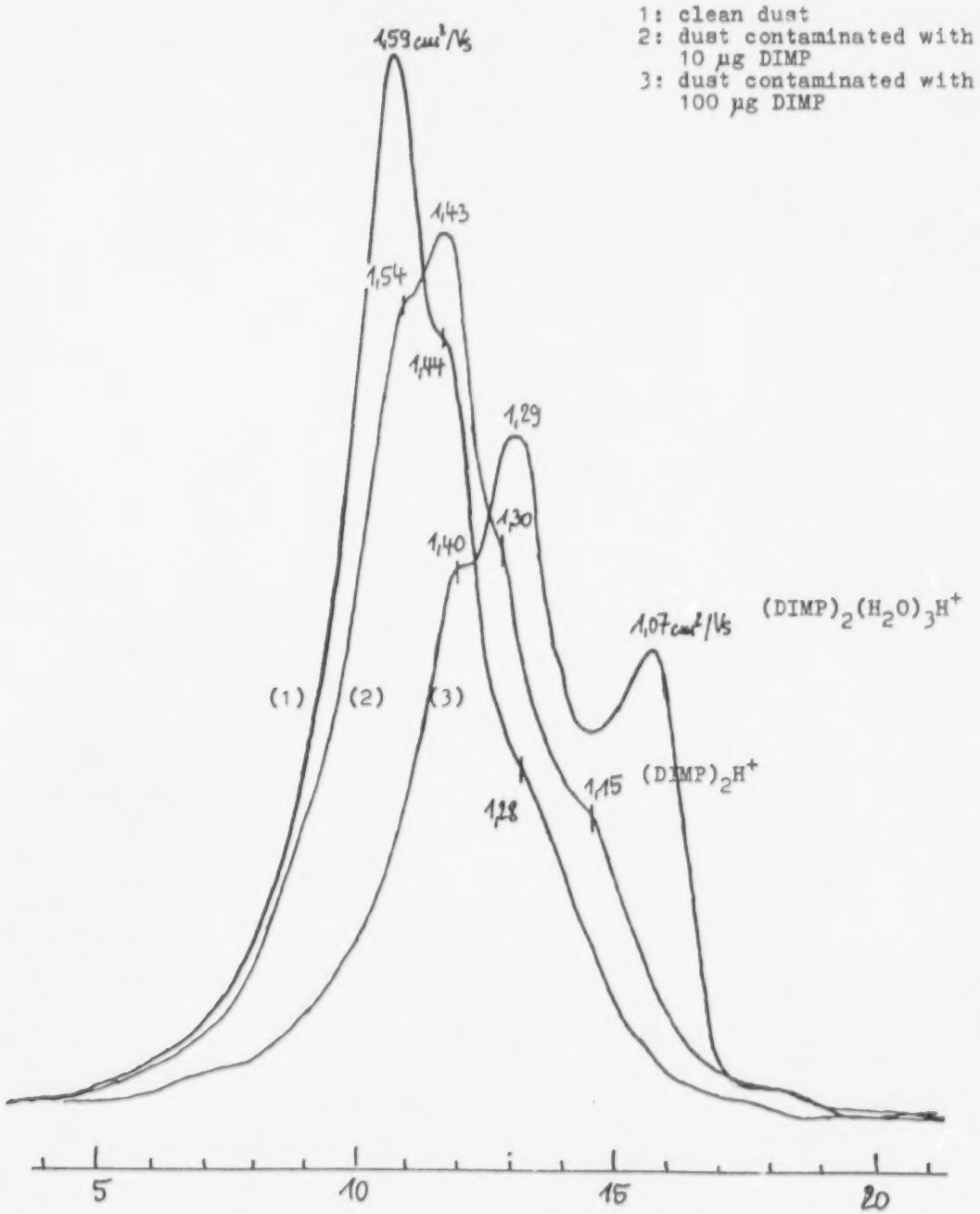
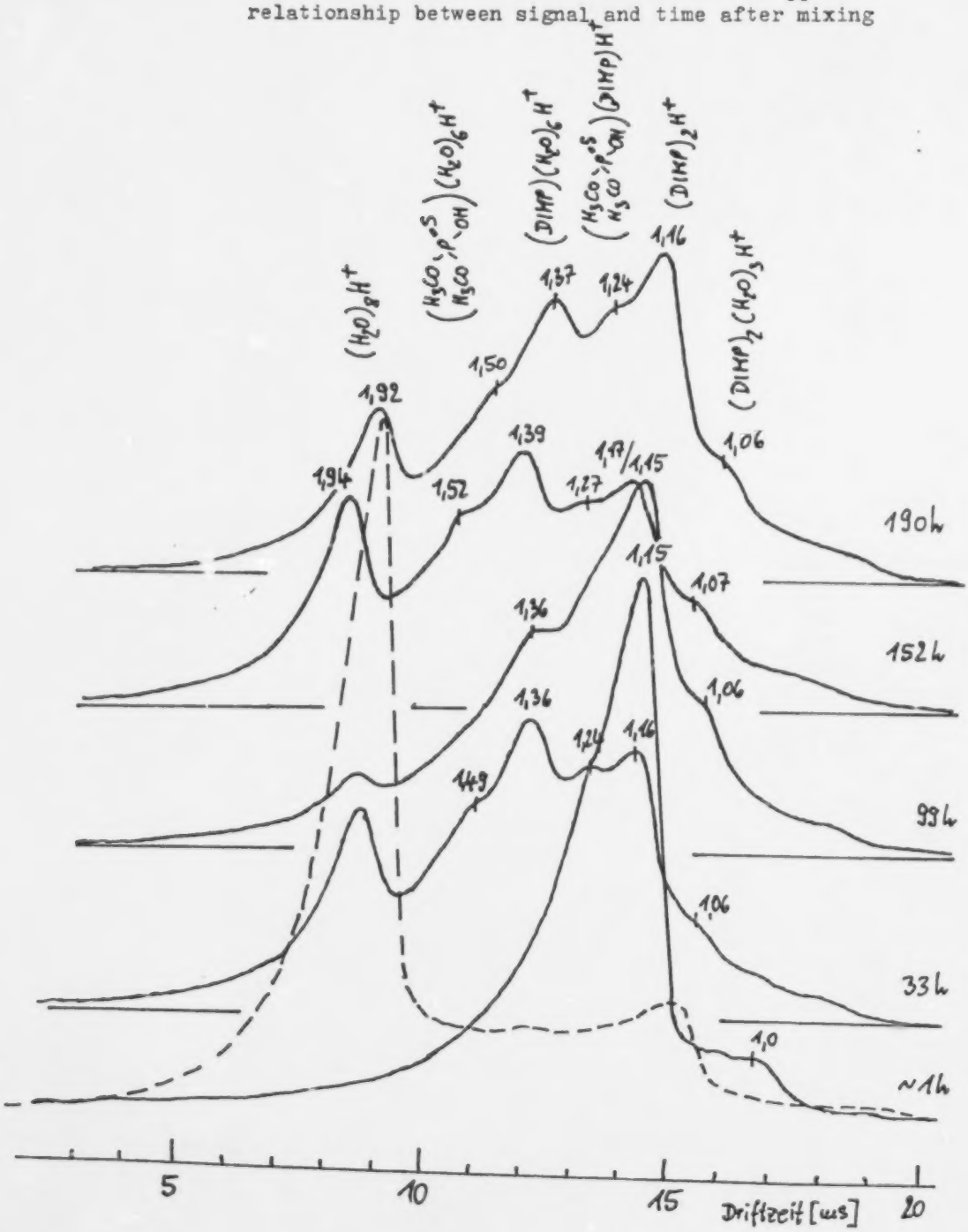


Fig. 15: IMS signals recorded at measurement point 8 (parathion-methyl, DIMP content was 0,4 $^{o}/_{oo}$), relationship between signal and time after mixing



CONFERENCE ON DISARMAMENT

CD/999
CD/CW/WP.295
12 June 1990

Original: ENGLISH

AUSTRIA

# Report on a National Trial Inspection

## 1. General remarks

The Austrian National Trial Inspection was conducted in order to actively contribute to the solution of outstanding technical problems for the elaboration of a global Convention on the Prohibition of the Use, Development, Stockpiling and Possession of Chemical Weapons and on their Destruction and thereby to facilitate the early conclusion of such a convention.

A preliminary report on the results of the Austrian National Trial Inspection was put forward to the Conference on Disarmament under document CD/948 (CD/CW/WP.260) dated 14 August 1989. The present comprehensive report contained in this paper is following the blueprint outlined in documents CD/CW/WP.213 and CD/CW/WP.248/Rev.1.

## 2. Objectives of the National Trial Inspection

The NTI was planned as a routine inspection in a multi-purpose plant. The facility inspected is part of this plant. It consumes a Schedule 2 chemical ("chemical A") for the production of a "chemical B" in a batch process. It was the objective of the conducted NTI to verify that:

(a) the Schedule 2 chemical has not been diverted for purposes prohibited by the Convention; this implied the verification of the initial declaration; and

(b) the non-production of chemicals listed in Schedule 1 within the facility inspected. The time selected for the routine inspection was shortly after the end of the work on a production batch. It was the main objective of the trial inspection to find out if compliance with the Convention can be verified at this time by checking the mass balance of the consumption of chemical A on the basis of administration records, technical administration

records, technical records of the production process and the results of samples drawn to check the identity of the product B. Likewise, it was the purpose to verify the non-production of Schedule 1 chemicals in the facility inspected by the absence of traces of these compounds.

3. Degree of realism of the National Trial Inspection

The conduct of a NTI in a multi-purpose production plant represents a realistic reflection of the structure of the Austrian chemical industry which is most relevant for the future Convention. Working out the facility attachment constituted an overall learning process by the technical inspection team, the persons responsible for the production process and the representatives of the Foreign Ministry involved.

The inspection team was separated into two groups which consisted of one analytical group and one technical group which inspected the equipment and the records of the company. In order to obtain independent views on the production process both teams worked independently during the inspection. The results obtained were only merged for the final report.

4. Selection of the team

The complexity of operation of a chemical plant comprising a larger number of different production facilities made it necessary to include in the inspection team experts on business administration techniques as used in modern chemical enterprises, chemical engineering, process control and analytical chemistry.

5. Selection of the facility

The facility selected was one of the very few Austrian facilities consuming a Schedule 2 chemical. It is housed in a building containing other facilities and situated at an industrial site where a number of chemical companies operate other multi-purpose plants. The site also comprises storage facilities and buildings which house administration and research laboratories.

6. Confidentiality of the National Trial Inspection

Confidentiality was one of the main subjects in the preparatory discussions during the initial visit with representatives of the company to be inspected. It was agreed that the inspectors should use, as far as feasible, non-confidential information. Confidential information contained in technical and business documents was only to be used on the inspection site. All participants were Austrian citizens and were obliged to keep confidentiality because of their official positions. In addition, each member of the

inspection team had to sign a special declaration to keep all information gained during the inspection confidential. This declaration was decisive for the readiness of the company to provide confidential information needed for working out the facility attachment and carrying out the inspection.

7. Conduct of the National Trial Inspection

During the initial visit which took place on 13 July 1989 an agreement based on CD/CW/WP.248 was reached between the representatives of the company and the inspection team on the inspection mandate. The discussions included the subject of confidentiality, size and composition of the inspection team and outlined the inspection procedures. The working out of the facility attachment according to the rolling text, version CD/881, pages 124-127, was followed by an introductory briefing on technical and administrative details by company representatives. Based on this information the inspection team defined the nature and extent of background material needed for the actual carrying out of routine inspections.

The exact definition of the material needed proved essential for the adequate preparation of the inspection by the administrative branch of the company and thereby reduced the amount of time needed for the inspection.

PRESENTATION OF THE RESULTS OF THE NATIONAL TRIAL INSPECTION

A. GENERAL APPROACH

1. Objective of the National Trial Inspection

The NTI had the aim as outlined in paragraph 4 of the Annex to article VI (2) of the rolling text CD/881. It was thus conducted in order to verify that:

- the quantities of the Schedule 2 chemical A processed in the facility were consistent with the needs for purposes not prohibited by the convention;
- the declared chemical was not diverted or used for purposes prohibited by the convention; and
- the facility was not used to produce any chemical listed in Schedule 1.

2. Respective provisions in the rolling text of the convention for the conduct of the National Trial Inspection

The National Trial Inspection was designed as a "routine inspection" with the aim to verify that the production processes and the uses of the facility corresponded to the information given by the company in its initial declaration. It was assumed that the NTI simulated one of the series of trial

inspections needed for the verification of the use of a Schedule 2 chemical and for the non-production of a Schedule 1 chemical in the facility inspected. The trial inspection was designed in a way that took into account the following two characteristics of the verification tasks:

(a) The facility inspected was one of several equivalent facilities situated at the same site; and

(b) The Schedule 2 chemical was processed in a batch production. In this context it was agreed that the inspection would be carried out in a time interval between two batch production cycles.

3. Type of the on-site inspection

In accordance with paragraph 5 of the Annex to article VI (2) of the rolling text, the trial inspection was prepared by a number of discussions with representatives of the plant and by an initial visit of the inspectors lasting one day. The initial visit was started by an extensive briefing on the organization of the production and the characteristics of the production process and of the equipment.

4. Advance information declaration

The initial declaration contained the following information:

- the exact definition of the Schedule 2 chemical A;
- the production process by which the chemical is processed;
- the uses of the chemical B produced.

5. Type of facility

The facility inspected was a multi-purpose production facility which is normally used for the synthesis of pharmaceutical products.

6. Type of declared activity at the facility

The facility is used to perform a chemical reaction in which batches of the Schedule 2 chemical A are transformed into the pharmaceutical product B. The final product is, to the largest extent, designated for export.

7. Activity at the facility during the inspection

The trial inspection took place between two production batches; at the time of the inspection the equipment was used for producing another chemical.

B. DETAILED APPROACH

8. The inspection mandate

In order to increase speed and efficiency of the inspection, the inspection team split up into two groups: an analytical group which collected and analysed samples and a technical group which inspected the equipment and

checked administrative and technical documentation. Both groups worked in parallel; their results were merged in a joint discussion shortly before the convening of the closing conference.

9. Composition of the inspection team

The inspection team included the following persons:

- One expert in chemical technology, Professor at the Technical University of Vienna;
- One expert in analytical chemistry, University of Vienna;
- One chemical expert working at the Office of Defense Techniques, Ministry for Defense;
- One representative of the Ministry of Economic Affairs, acting as chairman of the inspection team;
- One representative of the Division for Arms Control and Disarmament, Ministry for Foreign Affairs;
- One representative of the Austrian delegation to the Conference on Disarmament, Permanent Mission, Geneva;
- One representative of the Austrian Federation of Chemical Industries.

It was essential for the inspection team that the participating expert in chemical technology knew the plant from his previous work and at the same time had substantial knowledge of modern business administration techniques as used in today's chemical industry. In spite of this, the inspection could not have been carried out within the envisaged time schedule without the assistance of the technical and administrative representatives of the company inspected.

10. Inspection equipment

All the equipment needed was provided by the company; the analysis necessary for verifying the absence of a Schedule 1 chemical was carried out by another company which belongs to the same industrial group as the company inspected. The costs for the analysis were covered by the Ministry for Economic Affairs and the Ministry for Foreign Affairs.

11. Activities prior to the arrival of the inspection team at the site

All the documents necessary were listed up in advance and then provided by the company. They included the following:

(a) Information on the organization of the company, including maps indicating the location of the production and storage facilities;

(b) Information on the chemical reaction which consumes the Schedule 2 chemical A, including information on:

- flow-sheets (chemical formula flow-sheets, equipment flow-sheets and mass flow-sheets at each process step including quantitative information;
- standard operating procedures;
- documentation on the production program listing;
- quantities produced in the different production batches;
- documentation on the storage of the initial product A and the final product B;
- inventory transaction reports.

12. Advance preparation at the site

Advance preparation was limited to preparing access to the necessary updated documentation.

13. Escort and point of contact arrangements

At all stages of the inspection the inspectors were accompanied by representatives of the company which supported them in an extremely helpful and co-operative manner.

14. Additional participants

There were no additional participants present during the inspection.

15. Duration of the inspection and the initial visit

The duration of the initial visit was one day, the duration of the inspection was two days.

16. Measures to protect confidential information

All members of the inspection team were Austrian officials, and, therefore, obliged by law to keep information obtained confidential. In addition, the company requested that all participants signed a document which reaffirmed this obligation. It was agreed that administrative, commercial or technical documents would not be removed from the inspection site without the permission of the company. All documents should only be accessible to the members of the inspection team in the presence of a representative of the company.

17. Introductory Conference

In the course of the Introductory Conference, representatives of the company emphasized, already at this stage as well as repeatedly during the inspection, that the characteristics of the facility inspected would make it impossible to use it for the production of any Schedule 1 chemical.

In the course of the initial discussion, general agreement could be reached on the proposed inspection procedures. In addition to the facility attachment the company provided a plan of the production site indicating the location of other production and storage facilities as well as buildings housing the administration offices and various research laboratories. Information on the process consuming the Schedule 2 chemical A was provided in form of a flow-sheet which included more detailed information on the equipment used. Special emphasis was put on the information on legal constraints of the production processes. Likewise, the question of safety regulations applicable to the respective plant was discussed.

18. Types of records which may be required by the inspectors

The following types of records were identified:

- Administrative records: purchase requisitions, quotation requests, purchase orders, invoices, receiving reports, storage receipts, storage records, stock requisitions, shipment records, transfer records and waste disposal documents;
- Technical records: standard operating procedures, reaction schemes, equipment flow-sheets in case of multi-purpose plants and plant utilization plans.

19. Plant orientation tour

During the plant orientation tour the inspection team visited not only the inspected facility but also other facilities located in the same building, storage areas and the air control and waste water systems serving the inspected facility.

20. Inspection of areas and facility equipment

The verification of the non-production of a Schedule 1 chemical in the facility inspected was verified in the following ways:

(a) Inspection of the equipment with respect to its suitability for the synthesis of highly toxic compounds

Taking into account existing security standards in highly industrialized countries, a facility for the production of Schedule 1 chemicals requires sophisticated technical equipment in order to ensure safe operation. If such equipment is not present, it can be concluded that a facility is not used for the production of a Schedule 1 chemical.

The inspected facility was visited and its technical equipment inspected in detail. The facility is of standard technical design. No special equipment

as required for the handling or the production of highly toxic substances was found at the inspected site. No special safety precautions regarding highly-toxic substances for the operating personnel were available. No storage facilities for highly toxic chemicals were provided for and there was no equipment found for the disposal of highly toxic waste. The facility moreover is situated within a production complex comprising other production facilities. It was, therefore, concluded that the technical equipment present at the facility would not allow the production of highly toxic chemicals as listed in Schedule 1.

(b) Check of the production records of the facility to verify its use during the period under investigation

In principle, if a facility performs in accordance with the declaration by the company for the period under investigation and thereby makes full use of its capacities, any activity prohibited by the Convention can be excluded.

In order to verify that the products produced during the period under investigation corresponded to the declaration by the company, the log book of the facility and the records of its technical instrumentation were checked.

In spite of the fact that, due to the technical equipment of the facility, any production of highly toxic chemicals as listed in Schedule 1 could be excluded, the inspection team decided to investigate such a possible production by inspecting the production records of the facility. The log book and the recordings of the temperature, pressure and flows at various key points of the production facility were checked. The operting conditions as read from the recordings of the instrumentation of the facility were found to be in full agreement with the requirements of the production processes as declared by the company and as stated in the records of the log book.

It was, therefore, concluded that - during the period of investigation - the production facility was used as declared by the company and no undeclared production had taken place.

(c) Search for traces of Schedule 1 compounds resulting from prior clandestine production

In principle, it seems highly improbable that a facility which falls under a routine inspection scheme (Schedule 2 production) would be used for the production of a Schedule 1 chemical since there would be near certainty of detecting any such production. However, in the case of a modern multi-purpose plant allowing rapid shifts in production chains within the plant, the consideration of some additional aspects for the conduct of the inspection

seems to be useful. This holds specially true for industrial complexes comprising several multi-puprose facilities, as shifts of production between facilities are also feasible.

In such a case, the search for traces of clandestine production of a Schedule 1 compound should include all the relevant facilities of the production complex. As a logical consequence, a re-definition of the term "facility" as used by the Convention in order to include a multi-purpose production unit seems necessary. Likewise, it might be useful to define special sampling points located in common installations, serving more than the declared facility, in order to gain information on the presence or absence of a Schedule 1 chemical. Common air installation systems or waste water systems might serve as such adequate sampling points.

In spite of the fact that the equipment of the facility did not prove to be suitable for the production of highly toxic substances and that the production records had shown the correctness of the declaration of the company, it was decided to identify possible sampling points in order to gain experience for the possible detection of Schedule 1 compounds. In this context, two appropriate sample points were identified:

- At one sampling point dust from a central air filter was collected;
- At another sampling point a waste water sample from the main waste water outlet of the building, housing also other multi-purpose facilities, was taken.

The collection of these samples proved non-intrusive as it neither interfered with the actual production process nor did it provide any information on production technology used.

21. Verification of the non-diversion of the Schedule 2 chemical

(a) Check of the Schedule 2 chemical inventory

The Schedule 2 chemical under investigation is not produced by the company but bought from outside sources. If the inventory records concerning this chemical as reported by the company were correct, no amount of this chemical could be diverted or used for purposes prohibited by the Convention.

An input-output analysis covering a period of one year was established for the Schedule 2 chemical by carefully examining the following documents: purchase requisitions, quotation requests, purchase orders, invoices, receiving reports, storage receipts, storage records, stock requisitions, shipment records, transfer records and waste disposal documents.

At the time of the inspection, the current inventory of the Schedule 2 chemical was checked at the storage facility. The comparison of the input-output data for the chemicl concerned and the actual inventory proved to be corresponding with the consumption data given by the company. It could be concluded that the whole amount of the Schedule 2 chemical had been used for the production of the product B.

(b) Cross check of inventory and production records

Although the check of the overall production records, the Schedule 2 chemical inventory records and the production figures for chemical B corresponded with each other for the total period under investigation, a more detailed analysis can show if this correlation holds true for any given moment of this period. This would give additional proof that the data have not been tampered with.

Using quantitative production and shipment records for chemical B and all records relating to the Schedule 2 chemical, a cross check can determine if the correlation of data is consistent for any given moment of the period under investigation. Such a test was not actually executed during the trial inspection but it was found that the data available were sufficient to perform such a test.

(c) Conclusions of the verification of non-diversion of the Schedule 2 chemical

In summary it can be stated that a detailed examination of all production and inventory data for the Schedule 2 chemical proved that the facility had been used as declared by the company and that no amount of the Schedule 2 chemical had been diverted or used for any undeclared purposes.

22. Sample and sample taking procedures

In the course of the inspection, two types of samples were taken:

- Samples of the product B to confirm its identity and purity;
- Samples of dust from the central air filter and waste water outlet to verify the absence of traces of Schedule 1 chemicals.

All samples were taken by company personnel in the presence of an inspector at sampling points not known to the company in advance.

23. Handling of the samples

The samples for the identification and the check of the purity of the product B were transferred to the routine control laboratory for analysis. They were analysed during the inspection.

The samples for the verification of the absence of Schedule 1 chemicals were transported to the analytical laboratory of another company belonging to the same industrial group as the company inspected and analysed with sophisticated equipment and skills necessary to complete this task, despite the fact that specific research samples ("standards") of Schedule 1 chemicals were not available.

24. Analysis of the samples

The samples of the product B were analysed in the presence of an inspector in the plant central production control laboratory by thin layer chromatography. The results corresponded to the product specifications.

The dust and waste water samples were analysed for 14 Schedule 1 chemicals (Sarin, Soman, Tabun, VX, HN-1, HN-2, HN-3, Lewisit 1, Lewisit 2, Lewisit 3, Mustard Gas, Sesquimustard, O-Mustard and BZ). The measures applied gas chromatographic retention indexes in two stationary phases of widely differing polarity with FID, NPD and mass spectrometric detection using mass selective plots after electron impact and chemical ionization. All results obtained were negative.

25. Documentation of the inspection

All documents and data provided by the company remained at the inspection site with the exception of certain documents identified by company representatives.

26. Evaluation by the inspectors

The co-operation shown by the company representatives proved to be essential for the achievement of the inspection goal.

27. Closing Conference

The closing Conference was devoted to a discussion of the inspection findings with the representatives of the company and to the drafting of the "Preliminary Report on an Austrian National Trial Inspection". The completion of the final report was delayed since the results of the off-site analysis of samples were not immediately available. The report was finalized with the consent of the company.

28. Anomalies, disputes and complications

It was pointed out by the inspectors that verifying the material balance of consumption for a Schedule 2 chemical could be simplified by including an additional routine sampling point. The representatives of the company emphasized that such a control point could be easily established.

29. Report by the inspectors

The splitting up of the inspection team into two groups working in parallel - one analytical and one technical group - proved to be efficient not only in reducing the total inspection time but also the time demand on company personnel.

30. Impact of the inspection of the facility operation

The inspection had no impact on the routine operation of the plant.

31. Conclusions

(a) It was concluded that the conduct of a routine inspection in a non-intrusive way and within an acceptable time frame requires the full co-operation of the company inspected. This holds especially true for a multi-purpose plant which forms part of a larger industrial complex.

(b) In view of the fact that the documentation on the production and administration of chemical production complexes in highly industrialized countries form an interrelated net of information, it is practically impossible to hide any violation of the Convention conducted by a single person or a small group of persons. Such an attempt could only be initiated with the active support of the plant management. Also any attempt of falsifying the documentation would pose a major risk due to the extensive amount of time needed and the number of persons involved.

(c) It was emphasized that computerized methods of administration within modern chemical plants pose specific problems for inspecting a company only offering limited co-operation.

(d) It was identified as useful to elaborate a computer program for assisting inspectors in carrying out cross checks of inventory records of Schedule 2 chemicals consumed with records of final production. The application of such examination procedures would require the handling of a large data base. As an additional advantage, such a computer program would guarantee that data fed into the program were not recorded in detail as the output would only identify irregularities and thereby assure confidentiality.

(e) It was also concluded that for the verification of the non-production of a Schedule 1 chemical it would not suffice to check the

production records of the facility equipment alone in case that production at the facility inspected had ceased for part of the period under investigation.

(f) The representatives of the facility inspected expressed a preference that routine inspections should only be carried out by national inspectors. International inspections should be limited to cases of claimed violations of the Convention (i.e. challenge inspections) or possible ad hoc inspections.

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